Table 5. *Deviations* (Å) *of atoms from the least-squares planes through the uracil base and ribose moiety, and torsion angles* (°) *about the furanose ring bonds*

Atoms not used in the calculation of the plane are indicated by asterisks.

Plane (I) $-0.7503X + 0.6555Y + 0.0857Z = 12.6052$
Plane (II) $-0.2438X - 0.8577Y - 0.4526Z = 2.0957$

	Plane (I)		Plane (II)
C(1′)	−0·007	N(1)	−0·039
C(2′)	0·525*	C(2)	0·037
C(3′)	0·006	N(3)	−0·043
C(4′)	−0·010	C(4)	−0·011
C(5′)	1·021*	C(5)	0·068
O(1′)	0·001	C(6)	−0·017
r.m.s. (Δ)	0·009	O(2)	0·040*
σ (r.m.s. Δ)	0·002	O(4)	−0·028
		r.m.s. (Δ)	0·041
		σ (r.m.s. Δ)	0·002

E.s.d.'s in the torsion angles are ~3°.

C(4′)–O(1′)–C(1′)–C(2′)	−22·7
O(2′)–C(1′)–C(2′)–C(3′)	34·2
C(1′)–C(2′)–C(3′)–C(4′)	−31·3
C(2′)–C(3′)–C(4′)–O(1′)	18·6
C(3′)–C(4′)–O(1′)–C(1′)	1·9

The molecular packing viewed down *b* is shown in Fig. 2. The molecules pack as chains running parallel to *c* with waters situated between the chains. There is partial overlap of bases belonging to molecules related by the twofold axis parallel to *a*. The degree of overlap is similar to that in uracil (Stewart & Jensen, 1967).

The amino N(3) atom of the base forms a hydrogen bond with the phosphate O(8) atom at a distance of 2·8 Å. The O(2′) and O(3′) hydroxyl atoms of the ribose moiety form hydrogen bonds with the phosphate O(8) and O(6) atoms respectively.

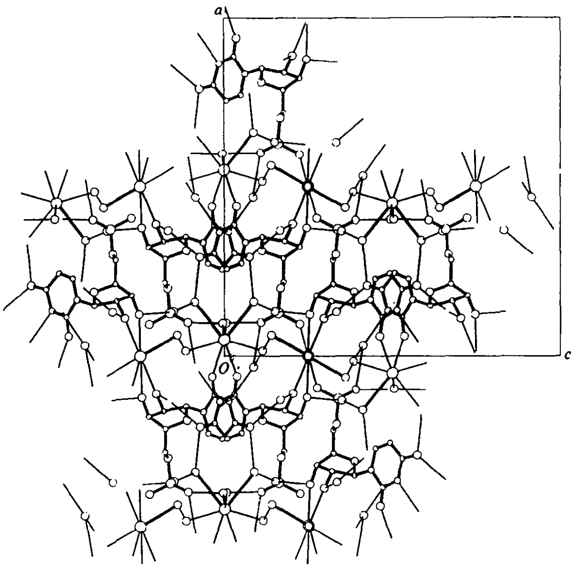

Fig. 2. Molecular packing viewed along *b*.

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Structure of Tetrakis[dichloroacetato-μ-(2-dimethylaminoethanolato)-copper(II)]

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Abstract. $C_{24}H_{44}Cl_8Cu_4N_4O_{12}$, $[Cu_4(C_2HCl_2O_2)_4(C_4H_{10}NO)_4]$, monoclinic, $C2/c$, $a = 18.675$ (9), $b = 15.109$ (8), $c = 16.403$ (8) Å, $\beta = 101.33$ (7)°, $V = 4538$ Å^3, $M_r = 1118.4$, $Z = 4$, $D_x = 1.638$ Mg m^{-3}, μ(Mo $K\alpha$) = 2·44 mm^{-1}. R is 0·050 for 1779 reflexions. The complex has a tetrameric cubane-type structure formed by four Cu and four bridging ethanolato O atoms with Cu—Cu distances of 3·124, 3·256, 3·609 and 3·935 Å.

Introduction. Blue crystals of the title compound were prepared by slow evaporation of an ethanol solution containing copper(II) dichloroacetate and 2-dimethylaminoethanol in a molar ratio of approximately 1:1.

0567-7408/80/040927-04$01.00

Intensity data were collected from a crystal 0·3 × 0·3 × 0·4 mm on a Syntex $P2_1$ four-circle diffractometer using Mo $K\alpha$ radiation and a graphite monochromator. Of 2581 independent reflexions measured with the ω-scan technique in the range $4° < 2\theta < 45°$, 1779 had $F > 5\sigma(F)$ and were used in the structure determination. The data were corrected for Lorentz and polarization factors and for absorption from φ-scan data.

The structure was solved by Patterson and Fourier techniques. Of the two possible space groups *Cc* and *C*2/*c*, the latter was chosen and confirmed as correct by the refinement. Scattering factors for neutral nonhydrogen atoms were from Cromer & Mann (1968) and those for H from Stewart, Davidson & Simpson (1965). Real and imaginary anomalous-dispersion corrections were included for the Cu and Cl atoms (Cromer & Liberman, 1970). The structure was refined by full-matrix least squares with anisotropic temperature factors for nonhydrogen atoms and isotropic temperature factors for H atoms. C—H vectors were constrained to lie in geometrically idealized orientations with C—H = 1·00 Å. The refinement converged to a conventional R value of 0·050 and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w(F_o)^2]^{1/2} = 0{\cdot}065$ with $w = 1/(30{\cdot}0 + |F_o| + 0{\cdot}02|F_o|^2)$. A final difference map showed no significant features. Final atomic coordinates are listed in Table 1, while interatomic distances and angles are given in Table 2. Computer programs used for the determination and refinement were those of XRAY 76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). A stereoview of the molecular structure is shown in Fig. 1.*

Table 1. *Fractional atomic coordinates* (×10^4)

	x	y	z
Cu(1)	−161 (1)	2354 (1)	−1451 (1)
Cu(2)	1048 (1)	1198 (1)	−2000 (1)
Cl(1)	1605 (3)	1869 (4)	1300 (3)
Cl(2)	1286 (3)	3706 (4)	922 (4)
Cl(3)	2909 (2)	2015 (2)	−4026 (3)
Cl(4)	3021 (3)	167 (3)	−3641 (3)
O(1)	−775 (3)	2378 (4)	−2540 (4)
O(2)	522 (5)	2557 (5)	−406 (4)
O(3)	1587 (4)	2030 (5)	−694 (5)
O(4)	136 (4)	1145 (4)	−1601 (4)
O(5)	1961 (4)	1145 (5)	−2403 (5)
O(6)	1498 (5)	1271 (8)	−3768 (5)
N(1)	−672 (5)	3506 (6)	−1251 (5)
N(2)	1237 (5)	−38 (5)	−1469 (5)
C(1)	−1295 (12)	3553 (14)	−1924 (10)
C(2)	−1303 (7)	3070 (8)	−2627 (8)
C(3)	−943 (8)	3434 (8)	−469 (9)
C(4)	−196 (9)	4273 (9)	−1191 (11)
C(5)	1195 (8)	2412 (7)	−255 (6)
C(6)	1631 (7)	2717 (9)	593 (6)
C(7)	546 (8)	−284 (8)	−1205 (8)
C(8)	166 (7)	517 (7)	−953 (7)
C(9)	1415 (8)	−684 (9)	−2075 (9)
C(10)	1850 (8)	1 (9)	−751 (9)
C(11)	1994 (7)	1178 (9)	−3166 (8)
C(12)	2789 (7)	1208 (9)	−3284 (7)

Table 2. *Interatomic distances* (Å) *and angles* (°)

Symmetry code: (i) $-x, y, -z - \frac{1}{2}$.

Cu(1)—Cu(2)	3·124 (2)	Cu(2)—Cu(1^{i})	3·256 (2)
—Cu(1^{i})	3·609 (2)	—Cu(2^{i})	3·935 (2)
—O(1)	1·924 (6)	—O(4)	1·942 (7)
—O(2)	1·949 (7)	—O(5)	1·947 (8)
—O(4)	1·938 (6)	—O(1^{i})	1·965 (6)
—N(1)	2·043 (9)	—N(2)	2·061 (8)
—O(1^{i})	2·633 (7)	—O(4^{i})	2·858 (6)
—O(6^{i})	3·063 (10)	—O(3)	2·517 (7)
O(1)—Cu(1)—N(1)	85·5 (3)	O(4)—Cu(2)—N(2)	84·8 (3)
—O(4)	91·9 (2)	—O(1^{i})	89·3 (3)
O(2)—Cu(1)—N(1)	88·3 (3)	O(5)—Cu(2)—N(2)	91·0 (4)
—O(4)	96·1 (3)	—O(1^{i})	94·9 (3)
O(1^{i})—Cu(1)—N(1)	118·5 (3)	O(4^{i})—Cu(2)—N(2)	110·8 (3)
—O(1)	76·3 (2)	—O(4)	71·2 (2)
—O(4)	72·0 (2)	—O(1^{i})	67·4 (2)
—O(2)	98·6 (3)	—O(5)	108·5 (3)
O(6^{i})—Cu(1)—N(1)	91·1 (3)	O(3)—Cu(2)—N(2)	94·9 (3)
—O(1)	76·6 (3)	—O(4)	88·0 (3)
—O(4)	76·8 (3)	—O(1^{i})	84·5 (2)
—O(2)	112·9 (3)	—O(5)	94·5 (3)
—O(1^{i})	137·5 (2)	—O(4^{i})	144·5 (2)
N(1)—C(1)	1·44 (2)	C(5)—O(2)	1·25 (2)
—C(3)	1·47 (2)	—O(3)	1·26 (2)
—C(4)	1·45 (2)	—C(6)	1·54 (1)
C(2)—C(1)	1·36 (2)	C(6)—Cl(1)	1·74 (1)
—O(1)	1·43 (1)	—Cl(2)	1·75 (1)
N(2)—C(7)	1·49 (2)	C(11)—O(5)	1·27 (2)
—C(9)	1·48 (2)	—O(6)	1·22 (1)
—C(10)	1·47 (2)	—C(12)	1·54 (2)
C(8)—C(7)	1·50 (2)	C(12)—Cl(3)	1·77 (1)
—O(4)	1·42 (1)	—Cl(4)	1·76 (1)
C(1)—N(1)—C(3)	108 (1)	O(2)—C(5)—O(3)	130 (1)
—C(4)	114 (1)	C(6)—C(5)—O(2)	117 (1)
—Cu(1)	105 (1)	—O(3)	113 (1)
C(1)—C(2)—O(1)	115 (1)		
C(3)—N(1)—C(4)	108 (1)		
C(7)—N(2)—C(9)	111 (1)	O(5)—C(11)—O(6)	129 (1)
—C(10)	111 (1)	C(12)—C(11)—O(5)	111 (1)
—Cu(2)	105 (1)	—O(6)	120 (1)
C(7)—C(8)—O(4)	107 (1)		
C(9)—N(2)—C(10)	109 (1)		
Cu(1)—N(1)—C(3)	109 (1)	C(5)—C(6)—Cl(1)	108 (1)
—C(4)	113 (1)	—Cl(2)	112 (1)
Cu(1)—O(1)—C(2)	112 (1)	Cl(1)—C(6)—Cl(2)	111 (1)
—Cu(2^{i})	113·7 (3)	Cu(1)—O(2)—C(5)	127 (1)
Cu(2^{i})—O(1)—C(2)	120 (1)		
Cu(2)—N(2)—C(9)	111 (1)	C(11)—C(12)—Cl(3)	111 (1)
—C(10)	110 (1)	—Cl(4)	109 (1)
Cu(2)—O(4)—C(8)	112 (1)	Cl(3)—C(12)—Cl(4)	108 (1)
—Cu(1)	107·2 (3)	Cu(2)—O(5)—C(11)	123 (1)
Cu(1)—O(4)—C(8)	120 (1)		

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34981 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

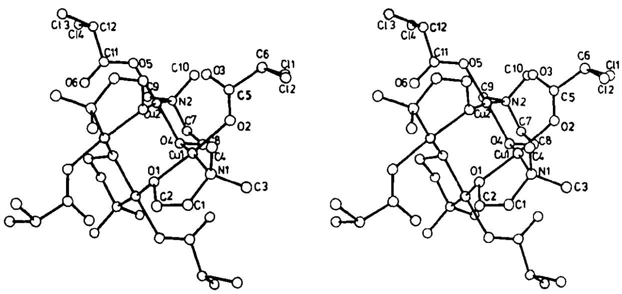

Fig. 1. Stereoview of the molecular structure.

Table 3. *Intermolecular contacts less than* 3·8 Å

Cl(1)···C(9^{ii})	3·29 (2)
C(6)···O(3^{iii})	3·32 (2)
Cl(3)···C(3^{iv})	3·56 (2)
Cl(3)···Cl(3^{v})	3·58 (1)
C(8)···C(8^{vi})	3·66 (2)
Cl(2)···O(5^{iii})	3·68 (1)
Cl(4)···C(2^{vii})	3·68 (1)
C(10)···Cl(4^{ii})	3·72 (2)
Cl(2)···C(4^{viii})	3·75 (2)
Cl(4)···C(1^{vii})	3·75 (2)

Symmetry code

(ii)	$x, -y, z + \frac{1}{2}$
(iii)	$-x + \frac{1}{2}, -y + \frac{1}{2}, -z$
(iv)	$x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$
(v)	$-x + \frac{1}{2}, -y + \frac{1}{2}, -z - 1$
(vi)	$-x, -y, -z$
(vii)	$x + \frac{1}{2}, y - \frac{1}{2}, z$
(viii)	$-x, -y + 1, -z$

Discussion. The structure consists of discrete tetrameric molecules, the shortest intermolecular distance being 3·29 Å between the Cl and methyl C atoms (Table 3). Each molecule may be considered as constructed around an eight-membered Cu_4O_4 ring of cubane-type structure. Within the Cu_4O_4 core the Cu—Cu distances vary between 3·124 and 3·935 Å, while the O atoms are in nearly tetrahedral array, with O—O distances 2·746 to 2·893 Å.

Each Cu atom is surrounded by two ethanolato O atoms, a carboxyl O atom and an amino N atom in a nearly square-planar arrangement with average Cu—O and Cu—N bonds of 1·944 and 2·052 Å. The Cu atoms lie almost in the least-squares planes calculated through equatorially coordinated atoms (Table 4). The axial sites of the Cu(1) atom are occupied by an ethanolato O atom and an O atom of the carboxylate group bonded to the adjacent Cu atom. The Cu—O distances are 2·633 and 3·063 Å, respectively. The corresponding Cu—O distances for the Cu(2) atom are 2·858 and 2·517 Å.

The symmetry of the molecules is C_2. This is higher than the point symmetry C_1 of $[Cu_4(Et_2NC_2H_4O)_4(Cl_2C_2HO_2)_4]$ (Smolander, Turpeinen & Ahlgrén, 1978) and $[Cu_4(Et_2NC_2H_4O)_4(Cl_3C_2O_2)_4]$ (Ahlgrén, Hämäläinen, Turpeinen & Smolander, 1979), but lower than the S_4 symmetry of $[Cu_4(Me_2NC_2H_4O)_4(ClC_2H_2O_2)_4]$ (Turpeinen, Ahlgrén & Hämäläinen, 1979) and $[Cu_4(Et_2NC_2H_4O)_4(ClC_2H_2O_2)_4]$ (Turpeinen, Hämäläinen, Ahlgrén & Smolander, 1979).

Table 4. *Least-squares planes and deviations* (Å) *of some atoms of interest from the planes*

Symmetry code: (i) $-x, y, -z - \frac{1}{2}$.

(*a*) Plane defined by N(1), O(1), O(4), O(2)

$$14{\cdot}793x + 6{\cdot}434y - 9{\cdot}585z = 2{\cdot}639$$

N(1)	−0·179 (9)	Cu(1)	0·027 (2)
O(1)	0·180 (6)	O(1^{i})	2·395 (6)
O(4)	−0·167 (6)	O(6^{i})	−2·476 (9)
O(2)	0·166 (8)		

(*b*) Plane defined by N(2), O(4), O(1^{i}), O(5)

$$5{\cdot}637x + 6{\cdot}329y + 12{\cdot}801z = -1{\cdot}227$$

N(2)	0·020 (6)	Cu(2)	0·016 (1)
O(4)	−0·021 (5)	O(3)	2·516 (6)
O(1^{i})	0·020 (5)	O(4^{i})	−2·476 (5)
O(5)	−0·019 (6)		

(*c*) Plane defined by O(2), O(3), C(5), C(6)

$$4{\cdot}623x + 13{\cdot}320y - 7{\cdot}260z = 3{\cdot}944$$

O(2)	−0·003 (7)	Cu(1)	0·170 (2)
O(3)	−0·002 (8)	Cl(1)	−1·657 (7)
C(5)	0·007 (11)	Cl(2)	0·918 (6)
C(6)	−0·002 (13)		

(*d*) Plane defined by O(5), O(6), C(11), C(12)

$$0{\cdot}214x + 15{\cdot}055y + 1{\cdot}312z = 1{\cdot}437$$

O(5)	0·013 (9)	Cu(2)	0·126 (2)
O(6)	0·014 (14)	Cl(3)	1·130 (4)
C(11)	−0·036 (17)	Cl(4)	−1·599 (6)
C(12)	0·009 (17)		

Angles between the planes (°)

(*a*)–(*b*)	88 (1)	(*a*)–(*d*)	67 (1)	(*b*)–(*d*)	61 (1)
(*a*)–(*c*)	42 (1)	(*b*)–(*c*)	84 (1)	(*c*)–(*d*)	32 (1)

The difference between the longest and shortest Cu—Cu distances in the present compound is 0·811 Å and is thus within the range of differences found for the complexes above (0·546–0·902 Å).

As well as the increasing distortion of the Cu_4O_4 core and the decreasing symmetry of the molecules on going from chloroacetate *via* dichloroacetate to trichloroacetate, the difference between the longest and shortest axial Cu—O distances also increases. The difference of 0·546 Å in the present compound is much greater than in $[Cu_4(Me_2NC_2H_4O)_4(ClC_2H_2O_2)_4]$ (0·025 Å) and $[Cu_4(Et_2NC_2H_4O)_4(ClC_2H_2O_2)_4]$ (0·057 Å) but slightly less than in $[Cu_4(Et_2NC_2H_4O)_4(Cl_2C_2HO_2)_4]$ (0·67 Å) where one Cu—O distance is as long as 3·26 Å. When the number of the chloro substituents is increased to three, as in $[Cu_4(Et_2NC_2H_4O)_4(Cl_3C_2O_2)_4]$, the one uncoordinated carboxyl O atom no longer lies in the axial position of the Cu atom.

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Dibromo[*N*-(2-pyridyl)acetamide]mercury(II)

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Abstract. $C_7H_8Br_2HgN_2O$, $[HgBr_2(C_7H_8N_2O)]$, monoclinic, $P2_1/c$, $a = 8{\cdot}070$ (1), $b = 8{\cdot}802$ (1), $c = 15{\cdot}942$ (3) Å, $\beta = 94{\cdot}13$ (1)°, $V = 1129{\cdot}5$ (5) Å^3, $Z = 4$, $M_r = 496{\cdot}56$, $d_x = 2{\cdot}92$ Mg m^{-3}, μ(Cu $K\alpha$) = 17·62 mm^{-1}. The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations. The final R was 0·077 for 2057 observed reflexions. The Hg atom is coordinated to two Br atoms, one O and one N. The ligand molecule bridges two Hg ions related by a twofold screw axis.

Introduction. Zinc(II), cadmium(II) and mercury(II) halide complexes with *N*-(2-pyridyl)acetamide were synthesized (Airoldi & Gonçalves, 1978). The structure determination of colourless crystals of the title compound was undertaken to study the coordination around the Hg^{II} atom. Cell dimensions were determined and refined by a least-squares fit to the setting angles of 25 reflexions on a CAD-4 automatic diffractometer. Intensity measurements were carried out up to $\theta = 75°$, using the ω–2θ scan mode, with graphite-monochromated Cu $K\alpha$ radiation and a prismatic crystal with a hexagonal base, approximated to a sphere of radius 0·2 mm. Lorentz, polarization and absorption corrections were applied (*International Tables for X-ray Crystallography*, 1972). Of the 2311 measured unique reflexions, 2057 were considered observed $[I > 3\sigma(I)]$* and retained for use in structure determination and refinement.

Table 1. *Fractional atomic coordinates for the mercury atom* (×10^5) *and for the other non-hydrogen atoms* (×10^4) *with their e.s.d.'s in parentheses*

	x	*y*	*z*
Hg	10220 (4)	30255 (4)	55333 (2)
Br(1)	3254 (2)	1588 (1)	4960 (1)
Br(2)	−1784 (2)	3996 (1)	5642 (1)
O	311 (9)	1065 (6)	6822 (4)
N(1)	−2725 (8)	−555 (8)	8360 (4)
N(2)	−840 (9)	−1005 (7)	7343 (4)
C(2)	−2309 (8)	−283 (8)	7588 (4)
C(3)	−3298 (11)	587 (9)	7021 (5)
C(4)	−4704 (12)	1222 (11)	7287 (7)
C(5)	−5110 (11)	979 (11)	8105 (6)
C(6)	−4142 (12)	57 (12)	8611 (5)
C(7)	333 (9)	−305 (8)	6930 (5)
C(8)	1668 (14)	−1276 (11)	6622 (6)

The structure was solved by the heavy-atom method. It was refined by full-matrix least-squares calculations by minimization of $\sum w(k|F_o| - |F_c|)^2$, where $w = (0{\cdot}016|F|^2 - 0{\cdot}516|F| + 6{\cdot}927)^{-1}$ for observed and $w = 0$ for unobserved reflexions, until all the atomic parameter shifts were smaller than each standard deviation. The final unweighted R factor, omitting unobserved reflexions, was 0·077, and including them 0·082. Anisotropic temperature factors were assigned to all non-hydrogen atoms. Complex neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974). The H atoms were placed at their calculated positions; their isotropic temperature factor was taken as 6·0 Å^2.

Positional parameters are given in Table 1.

Discussion. The complex is found to have an infinite extended chain-like structure with the organic ligand

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* Lists of structure factors, anisotropic thermal parameters, interatomic distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34973 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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