thermal parameters. Hydrogen atoms were placed in calculated positions and were assigned isotropic thermal parameters $U = 0.08$ Å^2. Final $R = 0.070$, $wR = 0.054$, maximum $\Delta/\sigma = 0.001$, $w = [\sigma^2(F)]^{-1}$. Maximum, minimum $\Delta\rho$ values in final difference synthesis 1.01, -0.52 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. Atomic fractional coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms are listed in Table 1.* Fig. 1. shows the atomic numbering scheme of the non-hydrogen atoms. Bond lengths and angles are given in Table 2. A view of the unit-cell contents is shown in Fig. 2. The structure contains two independent cations, each with crystallographically imposed $\bar{1}$ symmetry. Like $(C_{12}H_{25}NH_3)_2[ZnCl_4]$ (Ciajolo, Corradini & Pavone, 1977), the structure of DDAZn differs from that of $[NH_3(CH_2)_3NH_3][SnCl_4]$ (Kallet, Fall & Daoud, 1980) at room temperature in several ways. Crystals of $[NH_3(CH_2)_3NH_3][ZnCl_4]$ are monoclinic, space group $P2_1/n$ with $Z = 4$, whereas those of the title compound are triclinic, space group $P\bar{1}$ with $Z = 2$. As shown in Fig. 1 the cation containing C(6)—C(10) displays anomalous C—C bond lengths and U_{eq} values for its carbon atoms are physically unreasonable, probably as a result of disorder.

The tetrahedral $ZnCl_4^{2-}$ anions which form two-dimensional sheets parallel to 001 are sandwiched between hydrocarbon layers which consist of $[NH_3(CH_2)_{10}NH_3]^{2+}$ cations, as shown in Fig. 2. The average Zn—Cl bond length is 2.27 Å which accords with the corresponding value for $(n\text{-}C_{12}H_{25}NH_3)_2[ZnCl_4]$. All the Cl—Zn—Cl angles deviate by less than 4° fom 109.5° indicating a small distortion from tetrahedral symmetry. Since the shorter N···Cl distances range from 3.19 to 3.60 Å the $ZnCl_4$ tetrahedron may be connected to the 1,10-decanediammonium cation by medium to very weak N—H···Cl hydrogen bonds. However, the hydrogen bonds appear stronger in DDAZn than in $[NH_3(CH_2)_3NH_3][ZnCl_4]$ judging from the minimum N···Cl distance in each structure.

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Structure of a Copper Complex of an α-Hydroxylated Acid: $[Cu(C_{14}H_8O_3)(C_{12}H_8N_2)]_2.2H_2O$

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Abstract. Bis[μ-(9-hydroxy-9*H*-fluorene-9-carboxylato-*O*,μ-*O'*)]-bis(1,10-phenanthroline)copper(II)], $M_r = 971{\cdot}97$, monoclinic, $P2_1/n$, $a = 9{\cdot}101$ (2), $b = 20{\cdot}681$ (3), $c = 11{\cdot}101$ (1) Å, $\beta = 93{\cdot}55$ (2)°, $V = 2085$ Å^3, $Z = 2$, $D_x = 1{\cdot}55$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 11{\cdot}29$ cm^{-1}, $F(000) = 996$, $T = 297$ K, $R = 0{\cdot}039$ for 3064 observed reflections. Every Cu atom is square pyramidally coordinated by three O atoms and two N atoms. The two Cu atoms are connected to two bridging hydroxyl O atoms to

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form a Cu^{II} binuclear unit with a Cu_2O_2 core. The binuclear unit as a whole possesses a centre of symmetry with a Cu···Cu distance of 3·016 Å.

Introduction. Many copper(II) carboxylate complexes have been known for some time to have a quadruply bridged binuclear structure $[Cu(RCOO)_2L_2]_2$ (Doedens, 1976). In addition, some copper(II) complexes of α-hydroxylated acids have a mononuclear structure (Prout, Armstrong, Carruthers, Forrest, Murray-Rust & Rossotti, 1968; Ahlgrén & Hämäläinen, 1975, 1977; Ahlgrén, Hämäläinen & Pajunen, 1977). However, the crystal structure of $[Cu(C_{14}H_8O_3)(4,4'\text{-bipyridyl})]_2.DMF.H_2O$ has a binuclear unit with a Cu_2O_2 core which is the first representative of a new structure type in copper(II) carboxylate complexes (Liu, 1992). The title compound belongs to a series of $[Cu(\alpha\text{-hydroxylated } RCOO)L]_2$-type complexes studied by the present authors. In this paper the ligand L is 1,10-phenanthroline.

Experimental. Dark-blue prismatic crystals were grown from an aqueous solution. Cell dimensions and crystal-orientation matrix determined on an AFC-5R diffractometer by a least-squares treatment of the setting angles of 20 reflections in the range $10 < \theta < 15°$. Crystal dimensions 0·20 × 0·32 × 0·55 mm; intensities of reflections with indices h 0 to 11, k 0 to 26, l −14 to 14 with $2\theta < 55°$ measured; ω–2θ scans; ω-scan width $(1·575 + 0·400\tan\theta)°$; graphite-monochromatized Mo $K\alpha$ radiation. Intensities of three check reflections measured every 150 reflections showed no evidence of crystal decay. 5267 reflections measured, 4968 unique. 3064 with $I > 3\sigma(I)$ were used in structure solution and refinement; $R_{\rm int} = 0·011$. Data were corrected for Lorentz and polarization factors, and *DIFABS* absorption effect, maximum and minimum transmission factors 1·031, 0·936 (Walker & Stuart, 1983; Coppens, Leiserowitz & Rabinovich, 1965). Gaussian integration grid 6 × 6 × 6. Space group $P2_1/n$ uniquely determined from systematic absences.

The crystal structure was solved by the Patterson function method. Refinement was by full-matrix least-squares calculations, initially with isotropic and later with anisotropic thermal parameters. At an intermediate stage in the refinement, difference Fourier maps showed maxima in positions consistent with the expected locations of all H atoms; in the final round of calculations, the H atoms of a binuclear unit were positioned on geometrical grounds (C—H 0·95 Å) and all H atoms were included in the structure-factor calculations. The final cycle of refinement included 298 variable parameters, $R = 0·039$, $wR = 0·053$, goodness of fit = 1·39, $w = 1/\sigma^2(F_o)$. Maximum shift/e.s.d. = 0·03, and the final residual electron density was $+0·34$ ($-0·39$) e Å^{-3}; no chemically significant features. Scattering factors and anomalous-dispersion corrections for non-H atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV), and for H atoms from Stewart, Davidson & Simpson (1965). All calculations were made on a MicroVAX II computer using the *TEXSAN* software package (Molecular Structure Corporation, 1985). Atomic coordinates* and details of molecular geometry are given in Tables 1 and 2, respectively.

Discussion. The molecular configuration of the title compound is illustrated in Fig. 1; atoms labelled with 'a' are related by the centre of inversion to those listed in Table 1. The Cu atom is coordinated to two hydroxyl O atoms [O(1) and O(1^a)] of two acid ligands, one carboxyl O atom [O(3^a)] of one acid ligand, and two N atoms [N(2) and N(1)] of one 1,10-phenanthroline ligand to form a distorted square-pyramidal geometry; the Cu atom is shifted 0·2699 Å out of the basal plane towards the axial N atom [N(1)]. The axial Cu—N(1) bond is longer than normal axial Cu—N bonds (2·1 to 2·2 Å) found in five-coordinate Cu^{II} complexes with square-pyramidal configuration. Its length (2·304 Å) is also longer than the corresponding value (2·272 Å) in $[Cu(C_{14}H_8O_3)(4,4'\text{-bipyridyl})]_2.DMF.H_2O$ [complex (I)] (Liu, 1992). The longer Cu—N(1) bond might be caused by a steric effect. The two $[CuO_3N_2]$ square pyramids are paired by sharing a common edge O(1)—O(1^a) to form a binuclear unit $[Cu_2(C_{14}H_8O_3)_2(C_{10}H_8N_2)_2]$. The binuclear unit lies on a crystallographic centre at (0·5, 0, 0·5). The Cu—Cu^a distance of 3·016 Å and Cu—O—Cu^a angle of 102·4° are similar to the corresponding values in complex (I).

There are some common noteworthy features for both complex (I) and the title complex: (1) a binuclear configuration with a central planar Cu_2O_2 core; (2) a square-pyramidal coordination around the Cu atom; (3) the 9-hydroxy-9*H*-fluorene-9-carboxylic acid is coordinated to two Cu atoms through its carboxyl and hydroxyl O atoms, the hydroxyl O atom acting as a bridging atom to link up the two Cu atoms; the α-hydroxyl acid is a bivalent acid. This is quite different from most known copper(II) compounds of α-hydroxylated acids (Prout, Armstrong, Carruthers, Forrest,

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances and angles, intermolecular distances, least-squares-plane data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54384 (38 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HE0022]

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

$$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + 2ab(\cos\gamma)\beta_{12} + 2ac(\cos\beta)\beta_{13} + 2bc(\cos\alpha)\beta_{23}].$$

	x	y	z	B_{eq}
Cu	0·55608 (5)	0·01533 (2)	0·62750 (4)	2·34 (2)
O(1)	0·4550 (3)	0·0547 (1)	0·4845 (2)	2·6 (1)
O(2)	0·1704 (3)	0·1305 (1)	0·3051 (2)	4·0 (1)
O(3)	0·2845 (3)	0·0363 (1)	0·2940 (2)	3·0 (1)
O(4)	0·8894 (5)	0·1475 (2)	0·4026 (5)	8·8 (3)
N(1)	0·4080 (3)	0·0006 (1)	0·7863 (3)	2·9 (1)
N(2)	0·5886 (3)	0·0965 (1)	0·7220 (2)	2·5 (1)
C(1)	0·3254 (5)	−0·0471 (2)	0·8202 (4)	3·9 (2)
C(2)	0·2485 (5)	−0·0455 (3)	0·9265 (4)	4·6 (2)
C(3)	0·2571 (5)	0·0078 (3)	0·9978 (4)	4·5 (2)
C(4)	0·3597 (6)	0·1188 (3)	1·0332 (4)	4·5 (2)
C(5)	0·4466 (6)	0·1674 (2)	0·9980 (4)	4·4 (2)
C(6)	0·6212 (5)	0·2100 (2)	0·8544 (4)	3·9 (2)
C(7)	0·6957 (5)	0·2009 (2)	0·7530 (4)	3·9 (2)
C(8)	0·6772 (4)	0·1434 (2)	0·6887 (4)	3·3 (2)
C(9)	0·5136 (4)	0·1051 (2)	0·8228 (3)	2·5 (1)
C(10)	0·4196 (4)	0·0537 (2)	0·8581 (3)	2·6 (1)
C(11)	0·3444 (4)	0·0607 (2)	0·9644 (3)	3·4 (2)
C(12)	0·5277 (5)	0·1618 (2)	0·8927 (3)	3·3 (2)
C(13)	0·1445 (5)	0·0396 (3)	0·5922 (4)	4·4 (2)
C(14)	0·0446 (6)	0·0447 (4)	0·6846 (5)	7·2 (4)
C(15)	0·0306 (7)	0·1029 (5)	0·7435 (5)	8·7 (5)
C(16)	0·1090 (7)	0·1552 (4)	0·7159 (5)	6·9 (3)
C(17)	0·3526 (8)	0·2591 (3)	0·6231 (5)	7·1 (3)
C(18)	0·468 (1)	0·2909 (3)	0·5728 (7)	8·7 (4)
C(19)	0·5402 (8)	0·2624 (3)	0·4847 (5)	7·0 (3)
C(20)	0·5036 (6)	0·2007 (2)	0·4432 (4)	4·9 (2)
C(21)	0·3386 (4)	0·0998 (2)	0·4720 (3)	2·4 (1)
C(22)	0·2248 (4)	0·0928 (2)	0·5655 (3)	3·1 (2)
C(23)	0·2105 (5)	0·1515 (2)	0·6268 (4)	4·2 (2)
C(24)	0·3140 (5)	0·1970 (2)	0·5843 (4)	4·2 (2)
C(25)	0·3908 (5)	0·1686 (2)	0·4931 (3)	3·2 (2)
C(26)	0·2575 (4)	0·0895 (2)	0·3454 (3)	2·7 (1)

Table 2. *Bond lengths* (Å) *and angles* (°)

Cu—O(1)	1·962 (2)	Cu—O(1^a)	1·908 (2)	Cu—O(3^a)	1·962 (3)
Cu—N(2)	1·993 (3)	Cu—N(1)	2·305 (3)	O(1)—C(21)	1·411 (4)
O(2)—C(26)	1·225 (4)	O(3)—C(26)	1·271 (4)	N(1)—C(1)	1·309 (5)
N(1)—C(10)	1·357 (5)	N(2)—C(8)	1·327 (5)	N(2)—C(9)	1·358 (4)
C(1)—C(2)	1·409 (6)	C(3)—C(11)	1·414 (6)	C(4)—C(5)	1·351 (7)
C(6)—C(7)	1·363 (6)	C(7)—C(8)	1·392 (6)	C(9)—C(10)	1·434 (5)
C(13)—C(22)	1·365 (6)	C(14)—C(15)	1·38 (1)	C(16)—C(23)	1·397 (7)
C(17)—C(24)	1·393 (7)	C(18)—C(19)	1·35 (1)	C(20)—C(25)	1·369 (6)
C(21)—C(22)	1·517 (5)	C(21)—C(25)	1·515 (5)	C(21)—C(26)	1·561 (5)
C(22)—C(23)	1·401 (6)	C(24)—C(25)	1·395 (6)	Cu···Cu^a	3·016

O(1^a)—Cu—O(3^a)	83·3 (1)	O(1^a)—Cu—N(2)	169·5 (1)
O(1^a)—Cu—O(1)	77·6 (1)	O(1^a)—Cu—N(1)	112·9 (1)
O(3^a)—Cu—N(2)	98·2 (1)	O(3^a)—Cu—O(1)	151·9 (1)
O(3^a)—Cu—N(1)	92·3 (1)	N(2)—Cu—O(1)	97·1 (1)
N(2)—Cu—N(1)	77·5 (1)	O(1)—Cu—N(1)	114·0 (1)
C(21)—O(1)—Cu^a	115·3 (2)	C(21)—O(1)—Cu	131·7 (2)
Cu—O(1)—Cu^a	102·4 (1)	C(26)—O(3)—Cu^a	115·4 (2)
C(8)—N(2)—C(9)	118·7 (3)	C(9)—N(2)—Cu	118·5 (2)
Cu—N(1)—C(1)	133·8 (3)	Cu—N(1)—C(10)	108·3 (3)
C(1)—N(1)—C(10)	117·8 (3)	C(2)—C(3)—C(11)	119·3 (4)
C(7)—C(6)—C(12)	119·8 (4)	N(2)—C(9)—C(12)	121·9 (4)
N(1)—C(10)—C(11)	123·4 (4)	C(10)—C(11)—C(3)	116·8 (4)
C(6)—C(12)—C(9)	117·7 (4)	C(22)—C(13)—C(14)	118·3 (5)
C(15)—C(16)—C(23)	120·1 (6)	C(18)—C(19)—C(20)	121·6 (6)
O(1)—C(21)—C(22)	114·2 (3)	C(25)—C(21)—C(26)	113·1 (3)
C(16)—C(23)—C(24)	132·4 (5)	C(17)—C(24)—C(23)	131·5 (5)
C(20)—C(25)—C(21)	129·0 (4)	O(2)—C(26)—C(21)	119·6 (3)

Symmetry code: (a) $1-x$, $-y$, $1-z$.

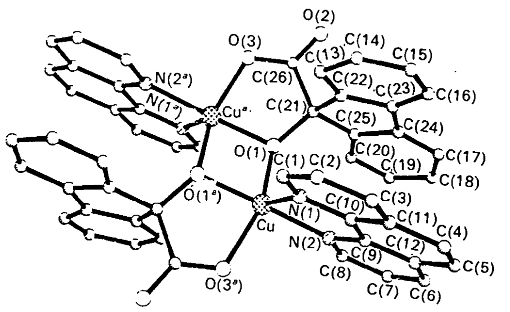

Fig. 1. Molecular structure of $[Cu(C_{14}H_8O_3)(C_{12}H_8N_2)]_2.2H_2O$.

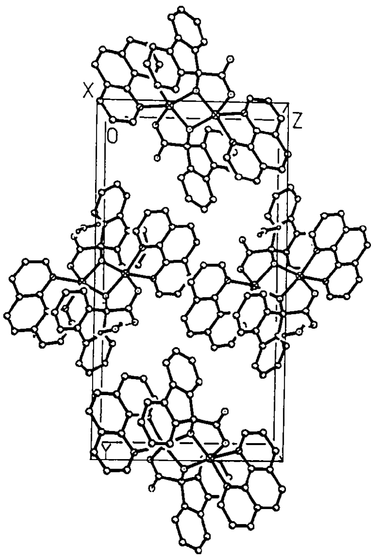

Fig. 2. A view of the molecular packing arrangement along the a axis.

Murray-Rust & Rossotti, 1968; Ahlgrén & Hämäläinen, 1975, 1977; Ahlgrén, Hämäläinen & Pajunen, 1977). However, there is an important difference between complex (I) and the title complex: in the former, molecules are connected together by the N atoms of 4,4′-bipyridyl ligands which then form an infinite network structure; in the latter, molecules are linked only by hydrogen bonds and van der Waals interactions, as shown in Fig. 2.

There is a weak intermolecular hydrogen bond O(4)—H(42)···O(2) between the O atom [O(4)] of a water molecule and the uncoordinated carboxyl O atom [O(2)], the O(4)···O(2) distance and O(4)—H(42)···O(2) angle being 2·860 Å and 165·6°, respectively.

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cis-Dichloro[2-(4-ethoxyphenyltelluro)ethyl methyl sulfide-*S*,*Te*]platinum(II), *cis*-$PtCl_2[Te(C_6H_4OC_2H_5)CH_2CH_2(CH_3)S]$

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Abstract. $[PtCl_2(C_{11}H_{16}OSTe)]$, $M_r = 589.9$, triclinic, $P\bar{1}$, $a = 9.185\,(4)$, $b = 11.179\,(2)$, $c = 8.396\,(2)$ Å, $\alpha = 103.82\,(2)$, $\beta = 105.49\,(3)$, $\gamma = 99.38\,(3)°$, $V = 783\,(1)$ Å^3, $Z = 2$, $D_m = 2.48$, $D_x = 2.50$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 11.346$ mm^{-1}, $F(000) = 540$, $T = 297\,(1)$ K, final $R = 0.0345$, $wR = 0.0371$ for 1937 unique observed reflections. The structure contains square-planar Pt with the Pt—Cl bond lengths being typical of those *trans* to a π-bonding ligand. The bond *trans* to Te is very slightly longer, 2.336 (3) Å, than that *trans* to S, 2.324 (4) Å. The Pt—Te bond is shorter, 2.514 (1) Å, than those reported previously where the Te atom was *trans* to another Te or to a P atom.

Introduction. An interest in the chemistry of Te compounds coupled with the recognition that, to our knowledge, there were only two reported structures of compounds containing a Pt—Te bond (Gysling & Luss, 1984; Kelly, Slawin, Williams & Woolins, 1990) led to this work describing the structure of *cis*-$PtCl_2[Te(C_6H_4OC_2H_5)CH_2CH_2(CH_3)S]$, a complex of a hybrid organotellurium ligand of (Te,S) type. Only recently has the ligand chemistry of a few multidentate hybrid Te donors been investigated (Singh, Srivastava & Khandelwal, 1990; Singh & Srivastava, 1990*a*,*b*). Like their P analogues, such ligands can generate interesting chemistry and the 2-(4-ethoxyphenyltelluro)ethyl methyl sulfide present in this Pt complex is one of the two examples of the (Te,S) type of ligand investigated to date.

Experimental. Preparation as described recently (Singh & Srivastava, 1990*c*) by reaction of $C_2H_5OC_6H_4TeCH_2CH_2SCH_3$ with K_2PtCl_4 in an acetone/water solution followed by slow evaporation of CH_3CN solution; density measured by flotation; crystal 0.31 × 0.47 × 0.37 mm; Rigaku AFC-6*S* diffractometer, highly oriented graphite monochromator; cell parameters from 25 strong reflections ($35.90 < 2\theta < 40.86°$); data collected at 297 (1) K using the ω–2θ scan technique to a maximum 2θ value of 50.0°; ω scans had an average width at half height of 0.31° with a take-off angle of 6.0°; scans of $(1.73 + 0.30\tan\theta)°$ at a speed of 32.0° min^{-1}. Weak reflections [$I < 10.0\sigma(I)$] rescanned (maximum of two rescans) and counts accumulated to assure good counting statistics. Diameter of incidence beam collimator was 0.5 mm and crystal to detector distance was 400.0 mm. Intensities of three representative reflections measured every 150 reflections changed by only 1.2% and a linear correction factor was applied; 2936 reflections; 2746 unique reflections ($4 < 2\theta < 50°$; $h = 0$ to 10, $k = -13$ to 13, $l = -9$ to 9). The linear absorption coefficient for Mo $K\alpha$ was 11.346 mm^{-1} and an empirical absorption correction, based on azimuthal scans of several reflections, was applied with transmission factors 0.5 to 1.0. The data were corrected for Lorentz and polarization effects. The structure was solved by direct methods (*SHELX*; Sheldrick & Egert, 1986). The non-H atoms, other than those in the phenyl ring, were refined anisotropically. The phenyl rings were constrained to a regular hexagon with bond distances of 1.40 Å and C—C—C angles of 120.0°. Phenyl and alkyl H atoms were also included in their idealized positions with C—H set at 0.95 Å and with the isotropic thermal parameters set at 0.007 Å^2 greater than that of the C atom to which they were attached. The final cycle of full-matrix least-squares refinement

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