

Fig. 3. Stereoview of the packing in the unit cell viewed down *a*.

Intramolecular distances in the range 2.90 (2)–3.02 (2) Å are observed between the amino-bonded Hg atoms and N(1) or N(7) (Table 3, deposited). These distances could indicate weak bonding interactions, but the angles are not favorable, the metal being far from the probable direction of the N(1) or N(7) lone pair: C(6)–N(1)–Hg(62) ave. 72, expected 120°; C(5)–N(7)–Hg(61) ave. 91, expected 129°.

A packing diagram is given in Fig. 3. The cell contains two independent pairs of centrosymmetrically related [(CH<sub>3</sub>Hg)<sub>2</sub>(mad)] molecules roughly perpendicular to the *ab* plane. Successive molecules along *c* are oriented 86° apart about the *c* direction and the

N(3) atom of one molecule is just above (or below) the mouth of the Y-shaped –N(HgCH<sub>3</sub>)<sub>2</sub> group of the next molecule, producing intermolecular N(3)···Hg contacts in the range 3.03 (2)–3.16 (2) Å (Table 2, deposited). In the *a* and *b* directions, all intermolecular contacts are of the normal van der Waals type.

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### Structure of Bis(diphenyl phosphato)tetrakis(imidazole)copper(II), [Cu{P(C<sub>6</sub>H<sub>5</sub>O)<sub>2</sub>O<sub>2</sub>}<sub>2</sub>(C<sub>3</sub>H<sub>4</sub>N<sub>2</sub>)<sub>4</sub>]

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(Received 4 September 1984; accepted 15 October 1984)

**Abstract.**  $M_r = 834.3$ , monoclinic,  $P2_1/c$ ,  $a = 8.264$  (3),  $b = 25.339$  (8),  $c = 10.216$  (3) Å,  $\beta = 114.82$  (4)°,  $V = 1941.6$  (8) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.43$  (1),  $D_x = 1.43$  g cm<sup>-3</sup>,  $\lambda(\text{Cu K}\alpha) = 1.5418$  Å,  $\mu = 21$  cm<sup>-1</sup>,  $F(000) = 862$ ,  $T = 295$  K. Final  $R = 0.033$  for 2284 unique observed reflections. The coordination of the Cu atom is tetragonally distorted octahedral with the basal plane defined by four coplanar imidazole N atoms and the axial sites occupied by unidentate diphenyl phosphato O atoms. The Cu atom lies very close to one

of the two unique coordinated imidazole ring planes [ $\Delta 0.049$  (1) Å] but is much more displaced [ $\Delta 0.104$  (1) Å] from the other one. The discrete uncharged molecular units are held together *via* N–H(pyrrole)···O(phosphate) hydrogen bonds.

**Introduction.** Studies on mixed-ligand complexes containing bipyridyl, histamine, histidine and imidazole (Mohan, Bancroft & Abbott, 1979; Antolini, Battaglia, Bonamartini Corradi, Marcotrigiano, Menabue, Pellacani & Saladini, 1982, and references cited therein) show that when metal ions are bound to these aromatic

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nitrogen donors they prefer to coordinate anionic ligands with oxygen donor sites rather than those which nitrogen ones.

Although some studies have been performed both in solution and in the solid state for copper(II)-imidazole-O ligands such as amino acids or N-protected amino acids, so far no structural information has been presented for copper(II)-imidazole-O(phosphate) complexes. We have now synthesized, isolated in the solid state and investigated by means of structural measurements, some new mixed-ligand complexes of copper(II) with imidazole and mono- and diaryl esters of phosphoric acid,  $(\text{ArO})\text{PO}_3\text{H}_2$ ,  $(\text{ArO})_2\text{PO}_2\text{H}$ , which were previously found to act as oxygen-donor anionic ligands (Narayanan, Ramirez, McCaffrey, Chaw & Marecek, 1978; Głowiak & Wnęk, 1984).

The results of the X-ray crystal-structure analysis of one of these complexes,  $[\text{Cu}(\text{ImH})_4(\text{DPhP})_2]$ , are described below [ $\text{ImH} = \text{C}_3\text{H}_4\text{N}_2$ ,  $\text{DPhP} = (\text{C}_6\text{H}_5\text{O})_2\text{PO}_2^-$ ].

**Experimental.** Barium diphenyl phosphate prepared by alkaline hydrolysis (with  $\text{BaCO}_3$ ) of diphenyl phosphoryl chloride obtained as described by Freeman & Colver (1938).

Excess ( $\times 8$ ) of imidazole (aq.) added to equimolar amounts of barium diphenyl phosphate and copper sulphate (anhydrous) dissolved in water. Evaporation of the filtered solution ( $\text{BaSO}_4$ ) produced beautiful light-violet-blue crystals; composition: calculated for  $\text{C}_{36}\text{H}_{36}\text{CuN}_8\text{O}_8\text{P}_2$ : C 51.82, H 4.36, N 13.43%; found: C 51.90, H 4.42, N 12.64%.  $0.15 \times 0.20 \times 0.25$  mm,  $D_m$  by flotation in benzene/ethylene dichloride; preliminary crystal data from oscillation and Weissenberg photographs; precise cell constants from setting angles of 15 reflections with  $24 < 2\theta < 34^\circ$ . Syntex P2<sub>1</sub> computer-controlled four-circle diffractometer, scintillation counter, graphite-monochromated Cu K $\alpha$  radiation; 2800 reflections measured,  $h$  0 to 8,  $k$  0 to 27,  $l$  -11 to 10,  $4.5 < 2\theta < 114.5^\circ$ ; of these 2284 with  $I > 3\sigma(I)$  considered observed and used in analysis; variable  $\theta$ - $2\theta$  scans, scan rate  $2.0$  to  $29.3^\circ \text{ min}^{-1}$  depending on intensity; two standard reflections (12 $\bar{3}$  and 251) monitored at intervals of 100 measurements, no significant fluctuations; corrections for Lorentz-polarization effects only. Structure solved by heavy-atom method and refined by full-matrix least squares [minimizing  $\sum w(|F_o| - |F_c|)^2$ , where  $w = 1/\sigma^2(F_o)$ ] with anisotropic temperature factors for non-H atoms; H atoms included in calculated positions with isotropic thermal parameters of  $4.80 \text{ \AA}^2$ , and C-H and N-H distances of  $1.00 \text{ \AA}$ . 46 atoms, 250 variables;  $(\Delta/\sigma)_{\text{max}} = 0.01$ ,  $\Delta\rho$  in final difference Fourier map within  $+0.20$  and  $-0.28 \text{ e \AA}^{-3}$ . Final  $R$ ,  $wR$  and  $S$  0.033, 0.042 and 4.069, respectively. Scattering factors for neutral atoms and corrections for anomalous dispersion (for Cu and P) from *International Tables for X-ray*

*Crystallography* (1974). All calculations performed on a Nova 1200 minicomputer with the Syntex (1976) XTL/XTLE system.

**Discussion.** Final positional and equivalent isotropic thermal parameters for non-H atoms are listed in Table 1;\* a perspective view of the molecule with the numbering scheme is shown in Fig. 1.

The special position  $\bar{1}$ , (0,0,0), is occupied by the  $\text{Cu}^{2+}$  ion. Each imidazole molecule coordinates through the pyridine-type N atom, forming a nearly regular square around Cu. The N(1)-Cu-N(3) and N(1)-Cu-N(3<sup>i</sup>) angles deviate only slightly from  $90^\circ$ . Relevant bond distances and angles are given in Table 2.

The metal-nitrogen distances of  $2.018$  (3) and  $2.020$  (2)  $\text{Å}$  are quite normal and are comparable with those found in  $[\text{Cu}(\text{ImH})_4\text{SO}_4]$ ,  $[\text{Cu}(\text{ImH})_4(\text{ClO}_4)_2]$  and  $[\text{Cu}(\text{ImH})_4(\text{NO}_3)_2]$ , i.e.  $2.00$ - $2.02 \text{ Å}$ ,  $1.997$ - $2.007 \text{ Å}$  and  $2.008$ - $2.018 \text{ Å}$ , respectively (Fransson & Lundberg, 1972; Ivarsson, 1973; McFadden, McPhail, Garner & Mabbs, 1976).

For symmetry reasons the atoms Cu, N(1), N(3), N(1<sup>i</sup>) and N(3<sup>i</sup>) are coplanar; above and below this plane O atoms from two diphenyl phosphate molecules are symmetrically placed and complete the tetragonally distorted (elongated,  $4 + 2$ ) octahedral coordination. The Cu-O(phosphate)<sub>ax</sub> distances of  $2.556$  (2)  $\text{Å}$  are considerably greater than those found for Cu-O(phosphate)<sub>eq</sub> bonds in such crystal structures as  $[\text{Cu}(3'\text{-GMP})(\text{phen})(\text{H}_2\text{O})_2 \cdot 7\text{H}_2\text{O}]$ ,  $[\text{Cu}(5'\text{-CMP})(\text{dpa})(\text{H}_2\text{O})_2 \cdot 5\text{H}_2\text{O}]$  (dpa = 2,2'-dipyridylamine), and  $[\text{Cu}(\text{ATP})(\text{phen})_2 \cdot 7\text{H}_2\text{O}]$ , where the Cu-O distances are  $1.929$ ,  $1.94$  and  $1.97$  (average values), and  $1.919$ - $1.977 \text{ Å}$ , respectively (Wei, Fischer & Bau, 1978; Aoki, 1979; Sheldrick, 1981).

The imidazole rings are almost planar [the largest deviation being  $0.006$  (4)  $\text{Å}$ ] but they are not coplanar with Cu. As has been found for  $[\text{Cu}(\text{ImH})_5\text{MPhP} \cdot 4\text{H}_2\text{O}]$  (MPhP = monophenyl phosphate) (Głowiak & Wnęk, 1985) and  $[\text{Cu}(\text{ImH})_4(\text{ClO}_4)_2]$  (Ivarsson, 1973), the two independent imidazole ligands have significantly different orientations with respect to the basal  $\text{CuN}_4$  plane; interplanar angles  $60.3$  (5) and  $103.3$  (3) $^\circ$ .

The general features of the diphenyl phosphate group agree well with other diaryl phosphates whose structures have been determined. A comparison of the bond lengths and angles of the present structure with those found for glycinium diphenyl phosphate (Głowiak & Wnęk, 1984) and pentaquaahexa(diphenyl phosphato)-

\* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters, torsion angles and the results of the least-squares-planes' calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39910 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.

trimagnesium(II) (Narayanan *et al.*, 1978) shows that dimensionally there are only rather minor differences, and most of these are, as expected, in the phosphate group. The most noticeable difference appears in the O(3)—P—O(4) angle, 97.6 (1)° in the copper complex which is considerably smaller than the value found for the corresponding angle in glycinium diphenyl phosphate, 104.4 (2)°.

Table 1. Positional parameters, ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2$ ) with *e.s.d.*'s in parentheses

$$B_{\text{eq}} = \frac{1}{3} \sum_i B_{ii}$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}$
Cu	0	0	0	2.90 (4)
P	3045 (1)	1109 (1)	2305 (1)	2.55 (5)
O(1)	1378 (3)	819 (1)	1519 (2)	3.26 (16)
O(2)	4649 (2)	819 (1)	3318 (2)	2.93 (15)
O(3)	2737 (3)	1620 (1)	3113 (2)	3.76 (17)
O(4)	3492 (3)	1417 (1)	1120 (2)	3.21 (16)
N(1)	-1748 (3)	32 (1)	906 (2)	2.73 (18)
N(2)	-3345 (3)	298 (1)	2032 (3)	3.84 (22)
N(3)	1515 (3)	-492 (1)	1604 (2)	2.85 (18)
N(4)	3330 (3)	-785 (1)	3745 (2)	3.22 (19)
C(1)	-2243 (4)	444 (1)	1442 (3)	3.48 (24)
C(2)	-3588 (5)	-231 (2)	1842 (4)	4.70 (30)
C(3)	-2595 (4)	-391 (1)	1159 (3)	4.00 (26)
C(4)	2542 (4)	-360 (1)	2949 (3)	3.05 (23)
C(5)	2767 (5)	-1212 (1)	2859 (4)	4.37 (30)
C(6)	1658 (5)	-1027 (1)	1549 (3)	4.04 (28)
C(11)	2301 (4)	1609 (1)	4282 (3)	2.94 (22)
C(12)	1396 (4)	1199 (1)	4559 (4)	3.89 (26)
C(13)	935 (5)	1243 (2)	5717 (4)	5.04 (32)
C(14)	1367 (5)	1685 (2)	6566 (4)	5.29 (34)
C(15)	2278 (5)	2090 (2)	6283 (4)	4.95 (31)
C(16)	2763 (4)	2053 (1)	5147 (4)	4.01 (26)
C(21)	5085 (4)	1662 (1)	1341 (3)	3.06 (22)
C(22)	5372 (5)	1731 (1)	111 (3)	4.23 (27)
C(23)	6886 (6)	1991 (2)	223 (5)	6.06 (36)
C(24)	8127 (5)	2172 (2)	1536 (5)	6.28 (39)
C(25)	7835 (4)	2095 (1)	2751 (4)	4.75 (31)
C(26)	6298 (4)	1844 (1)	2662 (3)	3.88 (26)

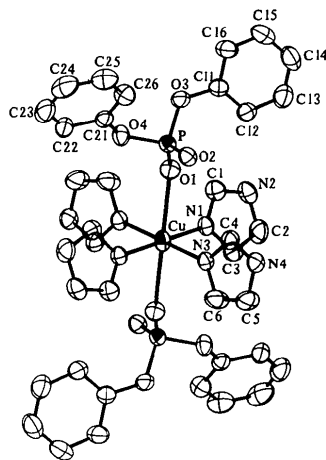


Fig. 1. ORTEP drawing (Johnson, 1976) of the molecule.

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

Cu—O(1)	2.556 (2)	Cu—N(3)	2.020 (2)
Cu—N(1)	2.018 (3)		
O(1)—Cu—N(1)	84.9 (1)	O(1)—Cu—N(3)	87.6 (1)
O(1)—Cu—N(3)	92.4 (1)	N(1)—Cu—N(3)	88.4 (1)
O(1)—Cu—N(1)	95.1 (1)	N(1)—Cu—N(3)	91.6 (1)
P—O(1)	1.467 (2)	P—O(3)	1.613 (2)
P—O(2)	1.488 (2)	P—O(4)	1.608 (2)
P—O(1)—Cu	144.6 (1)	O(1)—P—O(4)	106.9 (1)
O(1)—P—O(2)	119.4 (1)	O(2)—P—O(3)	109.0 (1)
O(1)—P—O(3)	111.6 (1)	O(2)—P—O(4)	110.1 (1)
		O(3)—P—O(4)	97.6 (1)
O(3)—C(11)	1.384 (4)	O(4)—C(21)	1.386 (4)
C(11)—C(12)	1.377 (5)	C(21)—C(22)	1.384 (5)
C(12)—C(13)	1.391 (5)	C(22)—C(23)	1.375 (6)
C(13)—C(14)	1.370 (5)	C(23)—C(24)	1.381 (6)
C(14)—C(15)	1.373 (6)	C(24)—C(25)	1.374 (6)
C(15)—C(16)	1.381 (5)	C(25)—C(26)	1.390 (5)
C(16)—C(11)	1.383 (4)	C(26)—C(21)	1.379 (4)
P—O(3)—C(11)	125.4 (2)	P—O(4)—C(21)	126.7 (2)
O(3)—C(11)—C(12)	123.6 (3)	O(4)—C(21)—C(22)	115.0 (3)
C(11)—C(12)—C(13)	118.7 (3)	C(21)—C(22)—C(23)	118.8 (3)
C(12)—C(13)—C(14)	120.8 (4)	C(22)—C(23)—C(24)	121.2 (4)
C(13)—C(14)—C(15)	119.9 (4)	C(23)—C(24)—C(25)	119.4 (4)
C(14)—C(15)—C(16)	120.3 (4)	C(24)—C(25)—C(26)	120.6 (4)
C(15)—C(16)—C(11)	119.5 (3)	C(25)—C(26)—C(21)	119.0 (3)
C(16)—C(11)—C(12)	120.8 (3)	C(26)—C(21)—C(22)	121.0 (3)
C(16)—C(11)—O(3)	115.6 (3)	C(26)—C(21)—O(4)	124.0 (3)
N(1)—C(1)	1.320 (4)	N(3)—C(4)	1.319 (4)
C(1)—N(2)	1.337 (4)	C(4)—N(4)	1.342 (4)
N(2)—C(2)	1.358 (5)	N(4)—C(5)	1.362 (4)
C(2)—C(3)	1.344 (5)	C(5)—C(6)	1.349 (5)
C(3)—N(1)	1.364 (4)	C(6)—N(3)	1.366 (4)
Cu—N(1)—C(1)	129.0 (2)	Cu—N(3)—C(4)	126.7 (2)
Cu—N(1)—C(3)	125.3 (2)	Cu—N(3)—C(6)	127.9 (2)
N(1)—C(1)—N(2)	111.0 (3)	N(3)—C(4)—N(4)	111.3 (3)
C(1)—N(2)—C(2)	107.2 (3)	C(4)—N(4)—C(5)	107.0 (3)
N(2)—C(2)—C(3)	106.7 (3)	N(4)—C(5)—C(6)	106.5 (3)
C(2)—C(3)—N(1)	109.5 (3)	C(5)—C(6)—N(3)	109.8 (3)
C(3)—N(1)—C(1)	105.6 (3)	C(6)—N(3)—C(4)	105.4 (3)

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

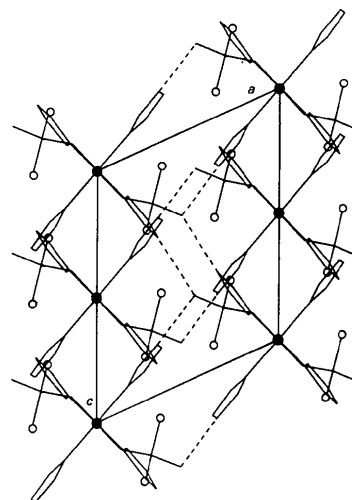


Fig. 2. The structure projected along *b* with the hydrogen-bonding scheme. Filled circles denote  $\text{Cu}^{2+}$  ions, open circles denote phenyl rings. Hydrogen bonds are indicated by dashed lines.

Drastic conformational changes occur in the diphenyl phosphate monoanion as it forms the copper complex. The torsion angles  $\omega$  and  $\omega^*$  of 61.4 (4) and 71.9 (4)° in the monoanion are -179.8 (2) and 77.0 (2)° upon coordination in the title compound.

Uncharged, discrete [Cu(ImH)<sub>4</sub>(DPhP)<sub>2</sub>] complex molecules are connected *via* hydrogen bonds from the pyrrole N atoms [N(2), N(4)] in the imidazole rings to the diphenyl phosphate O(2) atoms of neighbouring molecules, forming a two-dimensional network. Lengths and angles are 2.837 (4) Å, 163°, and 2.756 (3) Å, 170°, for the N(2)[H(2)]...O(2)( $x-1, y, z$ ) and N(4)[H(6)]...O(2)( $-x+1, -y, -z+1$ ) hydrogen bonds, respectively. A view of the unit-cell contents along **b**, with the hydrogen-bonding scheme, is shown in Fig. 2.

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\* Torsion angles O(3)-P-O(4)-C(21) and O(4)-P-O(3)-C(11) in the title compound.

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## Crystal Structures of Polyiodide Salts and Molecular Complexes. IX.\* Nonasodium Octa(*p*-toluenesulphonate) Triiodide Dihydrate, Na<sub>9</sub>(C<sub>7</sub>H<sub>7</sub>O<sub>3</sub>S)<sub>8</sub>I<sub>3</sub>·2H<sub>2</sub>O

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**Abstract.**  $M_r = 1993.24$ , triclinic,  $P\bar{1}$ , Dirichlet reduced cell used in structure analysis has  $a = 19.214$  (7),  $b = 18.526$  (7),  $c = 10.946$  (5) Å,  $\alpha = 88.69$  (8),  $\beta = 81.52$  (8),  $\gamma = 87.36$  (8)° [Delaunay reduced cell has  $a = 18.526$  (7),  $b = 19.214$  (7),  $c = 10.946$  (5) Å,  $\alpha = 98.48$  (8),  $\beta = 91.31$  (8),  $\gamma = 92.64$  (8)°],  $V = 3849$  (1) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.69$ ,  $D_x = 1.72$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 14.3$  cm<sup>-1</sup>,  $F(000) = 1980$ ,  $T = 298$  K,  $R_F = 0.089$  for 7975 measured reflections with  $F_o > 1.5\sigma(F_o)$ . There are alternating cationic (Na<sup>+</sup>, *p*-toluenesulphonates, waters) and anionic (non-interacting triiodides) sheets parallel to (010).

The methyl groups of the *p*-toluenesulphonate layers point away from the Na, O of the cationic layers, forming hydrophilic and hydrophobic layers. The overall arrangement of moieties in the crystal provides for local neutralization of charge. The compound is classified as a triiodide salt.

**Introduction.** Kastle & Hill (1894) prepared sodium and potassium salts containing both benzenesulphonate and triiodide anions. These are possible analogues to the various compounds of the herapathite series (Herapath, 1857; Jorgensen, 1877), which have quinine as cation and both sulphate (or selenate) and polyiodide anions. The structure of herapathite is not known although a preliminary crystallographic study has been reported (West, 1937).

\* Part VIII: Herbstein & Schwotzer (1984).

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