

Although the S₂P moiety is planar, the geometry about the P atom is distorted tetrahedral and as such would be expected to be sp³ hybridized; therefore, the electronic nature of the individual substituents on the P atom, R and R', would not be expected to effect one Ni–S bond in isolation, as has already been noted for at least three compounds listed in the *Introduction*. As a consequence, it would seem unlikely that the asymmetry found for (1) is electronic in nature.

Clearly there are no outstanding features in the structure of Ni(ⁱPrdt)₂ that may account for the unusual coordination found for (1) since the presence of the two large isopropyl groups does not impose any obvious steric constraints and hence there is no apparent reason for the asymmetry observed for (1).

We thank the Commonwealth of Australia for a Post-Graduate Research Award to one of us (ERTT).

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Acta Cryst. (1985). **C41**, 324–327

Pentakis(imidazole)copper(II) Monophenyl Phosphate Tetrahydrate, [Cu(C₃H₄N₂)₅][P(C₆H₅O)₃].4H₂O

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(Received 13 April 1984; accepted 15 October 1984)

Abstract. $M_r = 648.2$, monoclinic, $P2_1/c$, $a = 14.134$ (5), $b = 9.229$ (3), $c = 24.150$ (8) Å, $\beta = 110.60$ (5)°, $V = 2948.8$ (9) Å³, $Z = 4$, $D_m = 1.46$ (1), $D_x = 1.460$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.89$ mm⁻¹, $F(000) = 1348$, $T = 301$ K, $R = 0.050$ for 3830 unique observed reflections. The Cu atom is coordinated to the five planar imidazole ligands in rather strongly distorted (4 + 1) symmetry, Cu–N distances 2.028 (4), 2.036 (4), 2.036 (5), 2.062 (4) and 2.230 (5) Å, respectively. The crystal structure is strongly stabilized by a three-dimensional network of hydrogen bonds of the N–H...O(phosphate), O–H...O(water) and O–H...O(phosphate) types.

Introduction. The imidazole group of the histidine side chain is one of the most important binding sites for copper(II) in many metalloproteins and metal–protein

complexes.† The effectiveness of the imidazole group in acting as a metal-binding site has been attributed to its great flexibility, its availability at physiological pH (pK ca 7.0), and its capacity to form both σ and π bonds with metal ions (Eichhorn, 1973).

Although the detailed structures of a number of bis-, tris-, tetrakis-, hexakis- and some polynuclear-(imidazole)–copper(II) complexes are known (McFadden, McPhail, Garner & Mabbs, 1976; Fransson & Lundberg, 1978; and references cited therein), no structural information has been presented for a pentakis(imidazole)copper(II) cation.

We now report the structure of this cation following an X-ray crystallographic study of the title compound to provide additional precise values for copper(II)–imidazole bond lengths.

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† A list of examples of such binding with appropriate references has been deposited (Table 3). See deposition footnote.

Our results confirm the earlier investigations of Sjöberg (1972), who predicted the existence not only of pentakis- but also of hexakis(imidazole) complex ions of copper(II) in aqueous solution.

Experimental. Phenyl dihydrogen phosphate was obtained by hydrolysis, in a large excess of water, of monophenyl phosphoryl dichloride prepared as described by Freeman & Colver (1938). Dark-blue, well defined plates from aqueous solution of copper carbonate, phenyl dihydrogen phosphate and imidazole (1:1:10 molecular ratio) by slow evaporation at room temperature. $0.35 \times 0.40 \times 0.30$ mm, density by flotation in benzene/ethylene dichloride; preliminary crystal data from oscillation and Weissenberg photographs, Syntex P2, computer-controlled four-circle diffractometer, scintillation counter, graphite monochromator; cell parameters by least-squares refinement of setting angles for 15 reflections; 4629 independent reflections measured, $2\theta_{\max} = 55.5^\circ$, variable θ - 2θ scans, scan rate from 2.0 to $29.3^\circ \text{ min}^{-1}$ depending on intensity, two standards (152 and $3,3,10$) every 100 reflections with average intensities and e.s.d.'s of 92613 (1328) and 113254 (1624) respectively; corrections for Lorentz-polarization and absorption (transmission factors 0.775-1.000) but not for extinction. 3830 reflections with $I > 3.8\sigma(I)$ used for structure determination and refinement (index range: h 0-18, k 0-12, l -30-28); atomic positions of all non-H atoms from Patterson and Fourier synthesis, full-matrix least-squares refinement, neutral-atom scattering factors and anomalous-dispersion corrections (for Cu and P) from *International Tables for X-ray Crystallography* (1974), all calculations performed with the Syntex (1976) XTL/XTLE system. Eight of all 33 H atoms from difference Fourier map, remaining H atoms from geometrical considerations; non-H atoms refined with anisotropic temperature factors, H atoms with fixed isotropic temperature factors (4.5 \AA^2) included but not refined. 74 atoms, 370 variables; $(\Delta/\sigma)_{\max} = 0.1$, max. and min. $\Delta\rho$ in final difference Fourier map $\pm 0.6 \text{ e \AA}^{-3}$, $R = 0.050$, $R_w = 0.053$, $S = 3.240$, function minimized $\sum w(|F_o| - |F_c|)^2$, with $w = 1/\sigma^2(F)$.

Discussion. Final positional and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 1.* The bond lengths and angles are given in Table 2.

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates and details of mean-plane calculations and hydrogen-bonding geometry together with Tables 3 and 4 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39829 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional ($\times 10^4$) and equivalent isotropic thermal parameters (\AA^2) for non-H atoms with e.s.d.'s in parentheses

	x	y	z	B_{eq}
Cu	2716 (1)	2468 (1)	209 (1)	2.05 (3)
P	8241 (1)	2308 (2)	2504 (1)	1.82 (9)
O(1)	7899 (3)	734 (4)	2398 (2)	2.61 (28)
O(2)	8784 (3)	2880 (4)	2115 (2)	2.70 (28)
O(3)	7415 (3)	3298 (4)	2542 (2)	2.71 (29)
O(4)	9085 (3)	2204 (4)	3177 (2)	2.22 (26)
O(5)	4336 (4)	2752 (6)	2594 (2)	6.81 (53)
O(6)	961 (3)	3037 (5)	2586 (2)	4.94 (41)
O(7)	5777 (4)	762 (6)	2464 (3)	7.28 (56)
O(8)	2645 (3)	1344 (5)	2703 (3)	5.94 (49)
N(11)	4264 (3)	3247 (5)	738 (2)	2.62 (35)
N(13)	5675 (4)	3590 (6)	1497 (2)	3.15 (40)
N(21)	2284 (4)	4211 (5)	-340 (2)	2.69 (35)
N(23)	1889 (4)	5709 (6)	-1101 (2)	2.22 (39)
N(31)	3095 (3)	1440 (5)	-426 (2)	2.11 (31)
N(33)	2972 (4)	469 (5)	-1279 (2)	1.43 (33)
N(41)	2821 (3)	576 (5)	681 (2)	4.22 (38)
N(43)	2876 (3)	-911 (5)	1404 (2)	2.77 (36)
N(51)	2047 (3)	3439 (5)	733 (2)	4.30 (38)
N(53)	1832 (4)	4421 (5)	1507 (2)	3.05 (38)
C(12)	4872 (4)	2723 (7)	1248 (3)	2.90 (44)
C(14)	5586 (5)	4739 (8)	1139 (3)	4.19 (59)
C(15)	4707 (4)	4539 (7)	668 (3)	3.31 (49)
C(22)	1976 (4)	4336 (7)	-933 (2)	3.20 (48)
C(24)	2135 (5)	6531 (7)	-610 (3)	4.33 (57)
C(25)	2368 (5)	5618 (6)	-139 (2)	3.53 (50)
C(32)	2491 (4)	802 (6)	-919 (2)	2.78 (44)
C(34)	3932 (5)	907 (9)	-1022 (3)	5.10 (64)
C(35)	4014 (4)	1512 (8)	-493 (2)	3.84 (53)
C(42)	2531 (4)	356 (7)	1145 (2)	3.36 (48)
C(44)	3401 (5)	-1554 (7)	1095 (3)	3.66 (51)
C(45)	3369 (5)	-655 (6)	649 (3)	3.35 (47)
C(52)	2505 (4)	3874 (6)	1292 (2)	2.84 (45)
C(54)	897 (4)	4336 (6)	1068 (3)	3.22 (47)
C(55)	1034 (4)	3741 (6)	596 (2)	2.61 (42)
C(61)	9517 (4)	3380 (6)	3519 (2)	1.94 (37)
C(62)	9917 (4)	3098 (6)	4129 (2)	2.37 (39)
C(63)	10375 (4)	4223 (6)	4512 (2)	2.63 (42)
C(64)	10419 (4)	5613 (6)	4303 (2)	2.81 (43)
C(65)	10023 (4)	5861 (6)	3694 (2)	2.57 (41)
C(66)	9582 (4)	4742 (6)	3301 (2)	2.22 (39)

The crystal structure of the title compound consists of discrete $[\text{Cu}(\text{ImH})_5]^{2+}$ ions (Fig. 1), MPhP (monophenyl phosphate) dianions and water molecules of crystallization.

Each imidazole molecule coordinates through the pyridine-type N atom, forming a rather strongly distorted square pyramid around the central metal ion. The four basal N atoms are essentially coplanar; deviations from their mean plane for N(21), N(31), N(41) and N(51) are 0.014 (5), -0.011 (4), 0.013 (5) and -0.014 (5) \AA , respectively. The Cu atom is significantly $|-0.210 (1) \text{ \AA}|$ out of this plane.

The Cu atom is not required to lie in the imidazole ring plane (Freeman, 1967) and, in fact, it is found to be displaced considerably from each of the five rings [displacements varying from $-0.330 (1)$ to $0.311 (1) \text{ \AA}$] except one, ImH(51), where displacement of Cu is only $-0.063 (1) \text{ \AA}$. For comparison, similar small deviations were found in the crystal structures of the polymeric $[\text{Cu}(\text{ImH})(\text{Im})_2\text{Cl}]$ and $[\text{Cu}(\text{ImH})_2\text{Cl}_2]$ complexes, where the corresponding values are 0.051 and 0.02 \AA respectively (Lundberg, 1972a,b). On the other hand a considerably larger deviation is observed

in the $[\text{Cu}(\text{ImH})_4\text{SO}_4]$ structure, where the Cu atom is 0.354 Å out of coplanarity with one of the two unique imidazole rings (Fransson & Lundberg, 1972). It is interesting to note that in the case of the more weakly coordinated axial ligands there is an even greater displacement from the ring plane, 1.261 Å, in $[\text{Cu}(\text{ImH})_6](\text{NO}_3)_2$ (McFadden, McPhail, Garner & Mabbs, 1975).

Table 2. Bond lengths (Å) and angles (°) with *e.s.d.*'s in parentheses

(a) Pentakis(imidazole)copper(II) cation

	<i>n</i> = 1	<i>n</i> = 2	<i>n</i> = 3	<i>n</i> = 4	<i>n</i> = 5
Cu—N(<i>n</i> 1)	2.230 (5)	2.036 (4)	2.028 (4)	2.062 (4)	2.036 (5)
N(<i>n</i> 1)—C(<i>n</i> 2)	1.320 (7)	1.348 (6)	1.333 (7)	1.336 (7)	1.336 (6)
C(<i>n</i> 2)—N(<i>n</i> 3)	1.345 (8)	1.323 (8)	1.314 (7)	1.335 (8)	1.333 (8)
N(<i>n</i> 3)—C(<i>n</i> 4)	1.346 (9)	1.345 (8)	1.340 (9)	1.360 (8)	1.375 (8)
C(<i>n</i> 4)—C(<i>n</i> 5)	1.371 (9)	1.360 (9)	1.361 (9)	1.346 (8)	1.340 (8)
C(<i>n</i> 5)—N(<i>n</i> 1)	1.384 (8)	1.376 (7)	1.367 (8)	1.392 (8)	1.380 (8)
Cu—N(<i>n</i> 1)—C(<i>n</i> 2)	127.0 (4)	132.0 (4)	128.7 (4)	127.5 (4)	126.5 (4)
Cu—N(<i>n</i> 1)—C(<i>n</i> 5)	127.0 (4)	123.3 (4)	125.6 (4)	126.4 (4)	127.5 (4)
C(<i>n</i> 5)—N(<i>n</i> 1)—C(<i>n</i> 2)	104.8 (5)	104.4 (5)	104.6 (5)	105.3 (5)	106.0 (5)
N(<i>n</i> 1)—C(<i>n</i> 2)—N(<i>n</i> 3)	111.7 (5)	111.6 (5)	112.2 (5)	110.9 (5)	110.5 (5)
C(<i>n</i> 2)—N(<i>n</i> 3)—C(<i>n</i> 4)	107.9 (5)	107.6 (6)	107.4 (5)	107.8 (5)	107.5 (5)
N(<i>n</i> 3)—C(<i>n</i> 4)—C(<i>n</i> 5)	106.2 (6)	107.4 (6)	107.2 (6)	107.4 (6)	107.0 (6)
C(<i>n</i> 4)—C(<i>n</i> 5)—N(<i>n</i> 1)	109.4 (6)	109.0 (6)	108.7 (6)	108.8 (6)	109.1 (5)
N(11)—Cu—N(21)	97.3 (2)	N(21)—Cu—N(41)	167.3 (2)		
N(11)—Cu—N(31)	96.7 (2)	N(21)—Cu—N(51)	87.6 (2)		
N(11)—Cu—N(41)	95.2 (2)	N(31)—Cu—N(41)	92.1 (2)		
N(11)—Cu—N(51)	94.5 (2)	N(31)—Cu—N(51)	168.6 (2)		
N(21)—Cu—N(31)	88.7 (2)	N(41)—Cu—N(51)	89.3 (2)		

(b) Monophenyl phosphate dianion

P—O(1)	1.524 (4)	C(62)—C(63)	1.390 (8)
P—O(2)	1.503 (4)	C(63)—C(64)	1.388 (8)
P—O(3)	1.511 (4)	C(64)—C(65)	1.395 (8)
P—O(4)	1.646 (3)	C(65)—C(66)	1.394 (7)
O(4)—C(61)	1.370 (6)	C(66)—C(61)	1.379 (7)
C(61)—C(62)	1.405 (7)		
O(1)—P—O(2)	115.5 (2)	C(61)—C(62)—C(63)	118.7 (5)
O(1)—P—O(3)	112.4 (2)	C(62)—C(63)—C(64)	121.2 (5)
O(1)—P—O(4)	100.8 (2)	C(63)—C(64)—C(65)	118.9 (5)
O(2)—P—O(3)	113.7 (2)	C(64)—C(65)—C(66)	120.9 (5)
O(2)—P—O(4)	106.6 (2)	C(65)—C(66)—C(61)	119.3 (5)
O(3)—P—O(4)	106.4 (2)	C(66)—C(61)—C(62)	120.9 (5)
P—O(4)—C(61)	124.3 (3)		

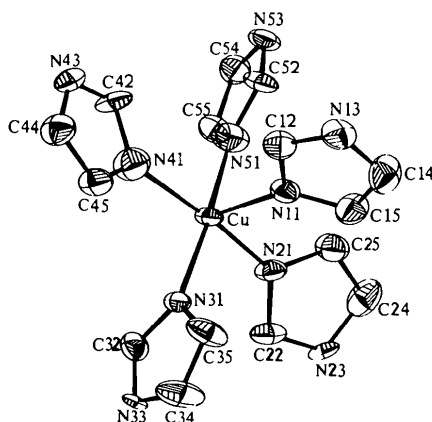


Fig. 1. ORTEP projection (Johnson, 1976) of the $[\text{Cu}(\text{ImH})_5]^{2+}$ ion with the atom-numbering scheme and thermal ellipsoids at the 50% probability level. H atoms have been omitted.

The orientation of the imidazole rings relative to the coordination square is variable in the various copper-imidazole complexes. In the title compound, for example, the imidazole ligands have significantly different orientations with respect to the basal plane, the angles ranging from 10.5 (6) to 110.0 (6)°. In contrast to this, all the imidazole rings in $[\text{Cu}(\text{ImH})_4(\text{NO}_3)_2]$ and $[\text{Cu}(\text{ImH})_4\text{I}_2]$ are very similarly oriented; in the first compound two unique imidazole ligands make angles of 94.6 and 98.6° respectively (McFadden *et al.*, 1976), while in the second one, all are approximately perpendicular to the equatorial plane (Akhtar, Goodgame, Rayner-Canham & Skapski, 1968). Thus the nature of the π -bonding between the imidazole N donor atoms and the Cu 3*d* orbitals will vary in these complexes.

The equatorial Cu—N distances (2.028–2.062 Å) are essentially equal (considering the experimental uncertainties, see Table 2), and are similar to the corresponding lengths found for other copper-imidazole complexes.* Although the mean values of their lengths fall into the 1.98–2.06 Å range, a value of 2.145 Å was found for the equatorial dimethylimidazole of the trigonal-bipyramidal complex dichlorotris(1,2-dimethylimidazole)copper(II) (Huq & Skapski, 1971).

The Cu—N(axial) distance of 2.230 (5) Å is considerably shorter than that reported for the distorted octahedral $[\text{Cu}(\text{ImH})_6]^{2+}$ cation [2.593 (3) Å] (McFadden *et al.*, 1975), but much longer than that observed in bis(dimethylglyoximate)(imidazole)copper(II) [2.141 (1) Å] (Morehouse, Polychronopoulou & Williams, 1980).

All five imidazole rings are planar [the largest deviation being 0.008 (7) Å] and their individual bond lengths and angles do not depart significantly from the respective mean values and correspond well with those found in free imidazole (X-ray, Martinez-Carrera, 1966; neutron diffraction, McMullan, Epstein, Ruble & Craven, 1979) and with those reported for the neutral ligand in other metal complexes.

The $(\text{C}_6\text{H}_5\text{O})\text{PO}_3^{2-}$ ion does not coordinate to the metal atom, but forms hydrogen bonds with the $[\text{Cu}(\text{ImH})_5]^{2+}$ ions and water molecules (see below). There are no unusual features in either the bond lengths or the bond angles in the anion (see Table 2).

An interesting aspect of this crystal structure is the hydrogen-bonding pattern. Discrete $[\text{Cu}(\text{ImH})_5]^{2+}$ ions are connected *via* hydrogen bonds from the pyrrole N atoms, N(*n*3), in the imidazole rings to the monophenyl phosphate dianion O atoms, forming a three-dimensional network. There are five such (crystallographically independent) hydrogen-bond contacts of different strengths and with parameters in the range:

* A table comparing Cu—N distances in copper-imidazole complexes with references has been deposited (Table 4). See deposition footnote.

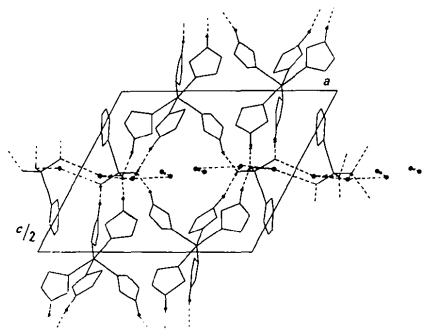


Fig. 2. Hydrogen-bonding scheme and packing viewed along *b*. H atoms from the organic ligands not involved in hydrogen bonding and those belonging to the water molecules have been omitted for clarity.

$2.683(6) < \text{N}-\text{H}\cdots\text{O} < 2.851(6) \text{ \AA}$ and $162 < \text{N}-\text{H}\cdots\text{O} < 176^\circ$. Two of them, $\text{N}(23)-\text{H}(23)\cdots\text{O}(2)(-x+1, -y+1, -z)$ and $\text{N}(33)-\text{H}(33)\cdots\text{O}(1)(-x+1, -y, -z)$, are very strong (especially the former) and nearly linear. Their $\text{N}\cdots\text{O}$ lengths, and angles at H being $2.638(6)$, $2.777(5) \text{ \AA}$ and 162 , 170° respectively. A similar short and nearly linear $\text{N}-\text{H}\cdots\text{O}(\text{P})$ hydrogen bond is observed in the crystal structure of imidazolium dihydrogen orthophosphate, where the corresponding values are $2.712(10) \text{ \AA}$ and 172° (Blessing & McGandy, 1972).

Of the atoms participating in hydrogen bonding only the water O atoms serve simultaneously as both acceptor and donor. There are four non-coordinated water molecules of crystallization, and all are involved in $\text{O}-\text{H}\cdots\text{O}(\text{water})$ and $\text{O}-\text{H}\cdots\text{O}(\text{P})$ hydrogen-bond formation. But while two of them, O(6) and O(8), are donors in two and acceptors in one such contact, water

molecule O(5) is a donor in two, and water molecule O(7) is a donor in only one hydrogen bond.

Fig. 2 shows the structure projected along *b* with the hydrogen-bonding scheme.

This work was supported by the Polish Academy of Sciences.

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Acta Cryst. (1985). **C41**, 327–329

Disordered Structure of Dibenzouranocene, $[\text{U}(\text{C}_{12}\text{H}_{10})_2]$

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(Received 20 July 1984; accepted 15 October 1984)

Abstract. $M_r = 546.46$, monoclinic, $P2_1/c$, $a = 9.524(4)$, $b = 8.558(4)$, $c = 11.758(6) \text{ \AA}$, $\beta = 113.52(4)^\circ$, $V = 878.7 \text{ \AA}^3$, $Z = 2$, $D_x = 2.065 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71073 \text{ \AA}$, $\mu = 88 \text{ cm}^{-1}$, $F(000) = 512$, $T = 296 \text{ K}$, $R = 0.052$ for 944 observed data. The uranium atom is sandwiched at the center (mean distance 1.91 \AA) of two planar and parallel [8]annulene

rings that are $\sim 6^\circ$ from an eclipsed conformation. The molecules pack in a disordered manner across a center of symmetry.

Introduction. The structures of a number of derivatives of uranocene, di[8]annuleneuranium(IV) ([8]annulene is cyclooctatetraene), have now been established