Pouchot & Guitel, 1980) by Day, Klemperer & Maltbie (1987): 1.29, 2.15 and 2.22 Å, which is consistent with the intermolecular hydrogen-bonding scheme.

The water molecule also links two polyanions with distances O(5)(O*f*2′)—O(*W*1) 2.88 (1) and O(*W*1)—O(12)(O*d*) (*x*, *y*, 1 + *z*) 2.98 (1) Å, the angle O(5)—O(*W*1)—O(12) being 135 (1)°.

The IR spectrum of this compound is very similar to the triply-protonated decavanadate of the same organic base (Arrieta, Gili & Lorente, 1984), except for three bands at 1100 (very strong), 1045 and 940 cm^{-1} (weak). According to Nakamoto (1986) these bands correspond to the Cl—O stretching frequencies of the perchlorate anion in a lower symmetry than *Td* symmetry.

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Structure of Tetrakis(imidazole)copper(II) Diiodide

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Abstract. $[\text{Cu}(\text{C}_3\text{H}_4\text{N}_2)_4]\text{I}_2$, $M_r = 589.67$, orthorhombic, $Pna2_1$, $a = 14.6703\ (8)$, $b = 9.4136\ (5)$, $c = 13.4665\ (8)$ Å, $V = 1859.7$ Å^3, $Z = 4$, $D_x = 2.11\ \text{g cm}^{-3}$, $\lambda(\text{Mo}\ K\alpha) = 0.71073$ Å, $\mu = 44.73\ \text{cm}^{-1}$, $F(000) = 1116$, $T = 295$ K, $R = 0.27$, $wR = 0.039$, 1668 unique reflections. The structure consists of discrete Cu(imH)_4^{2+} square-planar cations, where im is imidazole, and I^- anions which weakly interact with Cu in the fifth and sixth tetragonal positions. The N atoms of the imidazole rings which are not involved in bonding to the copper are arranged so that the point symmetry could contain a center of inversion. The six-coordinated species does not possess a center of inversion due to the different bonding distances of the two iodides and a moderate torsional twisting of one of the imidazole rings. The absolute configuration of the chiral complex has been determined using the η parameter [Rogers (1981). *Acta Cryst.* A**37**, 734–741].

Introduction. The tetragonal shape of this compound, with the copper–nitrogen and copper–iodide bond lengths, has been described in a preliminary communication (Akhtar, Goodgame, Goodgame, Rayner-Canham & Skapski, 1968). The bond angles, torsional distortion, relative positions of the second N atoms in the imidazole ring, the absolute configuration and crystal packing have not been reported. This paper presents the detailed structure of one enantiomer of this complex.

Experimental. $[Cu(C_3H_4N_2)_4]I_2$ was prepared by dissolving $[Cu(ClO_4)_2].6H_2O$ (3.7 g, 10 mmol) in 30 ml of absolute ethanol and adding 4.1 g of imidazole (60 mmol) with gentle heating. A 30 ml slurry of KI (3.3 g, 20 mmol) in absolute ethanol was added to this solution with stirring and heated to *ca* 333 K. Solid iodine (2.5 g, 20 mmol) was added with stirring and the solution filtered to remove the solid $KClO_4$. Crystals were obtained from the solution by slow evaporation of the solvent.

Symmetry and systematic extinctions ($0kl$, $k+l$ odd and $h0l$, h odd) indicated orthorhombic $Pna2_1$ or $Pnam$ as the choice for the space group. Successful refinement proved $Pna2_1$ to be correct. A crystal of approximate dimensions 0.1 × 0.3 × 0.5 mm was mounted on a glass fiber for data collection on an Enraf–Nonius CAD-4 diffractometer using a 1.6° θ–2θ scan with monochromated Mo $K\alpha$ radiation. Cell dimensions were determined by least-squares refinement of 25 reflections in the range $40 < 2\theta < 47°$. One standard reflection ($\overline{1}\overline{3}\overline{3}$) was monitored every 100 reflections and showed no systematic variation. Intensities of 1696 reflections were measured ($2\theta < 50°$, $0 < h < 16$, $0 \leq k \leq 16$, $0 \leq l \leq 11$) with 1668 unique reflections having $I < 3\sigma(I)$. ψ-scan absorption corrections were made.

The Cu and two I atoms were located using direct methods and subsequent cycles of structure-factor calculations and difference syntheses were used to locate the remainder of the non-H atoms. The origin was fixed by fixing the z parameter of the Cu atom. All non-H atoms were refined anisotropically. H-atom positions were calculated with isotropic temperature factors 30% greater than those of the atom to which they are bonded. Refinement continued, with the H atoms riding the C and N atoms and with their isotropic temperature factors held constant, to $R = 0.0266$ and $wR = 0.0391$ based on 207 least-squares parameters, and $w^{-1} = \sigma^2(F_o) + 0.00040|F_o|^2$, $\sum w(|F_o| - |F_c|)^2$ minimized. The goodness-of-fit was 1.87. The final shift to e.s.d. ratio was 0.05. The largest residual on the final difference map was 0.634 e Å^{-3}. The absolute configuration of the structure was determined with η-parameter [+0.83 (22)] refinement (Rogers, 1981) using *SHELXTL/PC* (Siemens Crystallographic Research Systems, 1990). All other data reduction, structure solution, refinement and graphics were executed using *MolEN* (Fair, 1990) on a VAX 6510 computer. Scattering factors and anomalous-dispersion corrections, provided by *MolEN*, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and Cromer & Mann (1968).

Table 1. *Positional parameters and equivalent isotropic thermal parameters* (Å^2) *with their e.s.d.'s for non*-H *atoms*

$$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cu*	0.12624 (4)	0.0117 (1)	0.68558	3.72 (2)
I1	0.14031 (3)	0.30607 (4)	0.83181 (5)	3.020 (8)
I2	0.11234 (3)	−0.32028 (4)	0.51741 (5)	3.191 (8)
N1	−0.0108 (3)	0.0186 (5)	0.6913 (4)	2.99 (9)
N2	0.1283 (3)	−0.1216 (6)	0.8037 (4)	3.0 (1)
N3	0.2613 (3)	−0.0087 (5)	0.6751 (4)	3.0 (1)
N4	0.1240 (3)	0.1313 (7)	0.5627 (5)	3.4 (1)
N5	0.1210 (4)	0.3126 (6)	0.4620 (6)	4.0 (1)
N6	0.1258 (4)	−0.3196 (6)	0.8851 (7)	4.4 (2)
N7	0.3959 (3)	−0.0571 (7)	0.6198 (6)	4.1 (1)
N8	−0.1504 (3)	0.0706 (7)	0.7291 (6)	4.3 (1)
C1	−0.0628 (4)	0.0992 (7)	0.7476 (5)	3.4 (1)
C2	−0.1564 (4)	−0.0256 (8)	0.6558 (7)	4.1 (1)
C3	−0.0697 (4)	−0.0583 (8)	0.6321 (5)	3.7 (1)
C4	0.1267 (4)	−0.2612 (8)	0.7962 (6)	4.0 (1)
C5	0.1280 (5)	−0.2117 (9)	0.9555 (6)	5.0 (2)
C6	0.1300 (5)	−0.0931 (8)	0.9016 (7)	4.8 (2)
C7	0.3232 (4)	0.0394 (7)	0.7427 (6)	3.8 (1)
C8	0.4070 (4)	0.0096 (8)	0.7083 (7)	4.6 (2)
C9	0.3081 (4)	−0.0642 (7)	0.6015 (5)	3.6 (1)
C10	0.1258 (5)	0.0834 (9)	0.4645 (6)	4.7 (2)
C11	0.1201 (4)	0.2671 (9)	0.5564 (6)	3.6 (1)
C12	0.1219 (6)	0.1991 (9)	0.4056 (8)	5.2 (2)

* z coordinate fixed.

Discussion. The final equivalent isotropic thermal and positional parameters of non-H atoms are listed in Table 1.* Bond lengths and angles are presented in Table 2. Packing of the ions in the unit cell is shown in Fig. 1. Fig. 2 shows one formula unit including the atom-numbering scheme. Fig. 3 shows how the imidazole rings are oriented with respect to the Cu—I directions.

The Cu—N distances of 1.996 to 2.026 Å fall in the normal range for the equatorial bond lengths in the tetragonal coordination of copper. These distances are comparable with those found in $[Cu(imH)_4SO_4]$, $[Cu(imH)_4(ClO_4)_2]$, $[Cu(imH)_4(H_2O)_2]F_2$, $[Cu(imH)_4\{P(C_6H_5O)_2O_2\}_2]$ and $[Cu(imH)_4(NO_3)_2]$, with values of 2.000–2.021, 1.998–2.010, 1.984–2.033, 2.018–2.020 and 2.008–2.018 Å, respectively (Fransson & Lundberg, 1972; Ivarsson, 1973; Vreugdenhil, Birker, ten Hoedt, Verschoor & Reedijk, 1984; Głowiak & Wnęk, 1985; McFadden, McPhail, Garner & Mabbs, 1976).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71103 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1017]

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

Cu—I1	3.406 (2)	Cu—I2	3.865 (2)
Cu—N1	2.013 (5)	N5—C11	1.34 (1)
Cu—N2	2.026 (5)	N5—C12	1.31 (1)
Cu—N3	1.996 (4)	N6—C4	1.32 (1)
Cu—N4	2.001 (7)	N6—C5	1.39 (1)
N1—C1	1.316 (8)	N7—C8	1.36 (1)
N1—C3	1.381 (8)	N7—C9	1.314 (8)
N2—C4	1.318 (9)	N8—C1	1.336 (8)
N2—C6	1.35 (1)	N8—C2	1.34 (1)
N3—C7	1.364 (8)	C2—C3	1.348 (9)
N3—C9	1.314 (8)	C5—C6	1.33 (1)
N4—C10	1.40 (1)	C7—C8	3.449 (9)
N4—C11	1.28 (1)	C10—C12	1.35 (1)
I1—Cu—I2	179.31 (3)	I1—Cu—N2	92.8 (2)
I1—Cu—N1	90.7 (2)	I1—Cu—N4	91.2 (3)
I1—Cu—N3	93.4 (2)	I2—Cu—N2	87.7 (2)
I2—Cu—N1	89.8 (2)	I2—Cu—N4	88.3 (2)
I2—Cu—N3	86.2 (2)	C10—N4—C11	105.1 (7)
N1—Cu—N2	90.3 (2)	C11—N5—C12	106.8 (7)
N1—Cu—N3	175.9 (2)	C4—N6—C5	108.3 (6)
N1—Cu—N4	89.8 (2)	C8—N7—C9	107.8 (6)
N2—Cu—N3	88.9 (2)	C1—N8—C2	109.7 (6)
N2—Cu—N4	176.0 (2)	N1—C1—N8	109.5 (6)
N3—Cu—N4	90.7 (2)	N8—C2—C3	105.4 (6)
Cu—N1—C1	128.3 (4)	N1—C3—C2	109.5 (6)
Cu—N1—C3	125.8 (4)	N2—C4—N6	110.3 (7)
C1—N1—C3	105.8 (5)	N6—C5—C6	104.0 (8)
Cu—N2—C4	123.9 (5)	N2—C6—C5	111.5 (7)
Cu—N2—C6	130.2 (5)	N3—C7—C8	108.0 (7)
C4—N2—C6	105.8 (6)	N7—C8—C7	106.9 (6)
Cu—N3—C7	125.6 (4)	N3—C9—N7	110.5 (6)
Cu—N3—C9	127.6 (4)	N4—C10—C12	107.2 (7)
C7—N3—C9	106.8 (5)	N4—C11—N5	112.4 (7)
Cu—N4—C10	126.9 (5)	N5—C12—C10	108.5 (9)
Cu—N4—C11	128.1 (6)		

The copper–iodide interactions are weak (3.406 for I1 and 3.865 Å for I2) but are definitely in the fifth and sixth coordination sites as indicated by the angles around copper (I1—Cu—I2 179.3; I1—Cu—N1,N2,N3,N4 from 90.7 to 93.4; I2—Cu—N1,N2,N3,N4 from 86.2 to 89.8°). The only known copper(II)–iodide distances are for five-coordinate complexes and these range from 2.672 to 2.718 Å for compounds such as $[\mathrm{Cu(phen)_2I]I.H_2O}$ (Nagle & Hathaway, 1991; Hambley, Raston & White, 1977; Hathaway & Murphy, 1980; Barclay, Hoskins & Kennard, 1963).

Bond distances and angles within the imidazole rings range from 1.314 (8) to 1.40 (1) Å and from 105.1 (7) to 112.4 (7)°, which are comparable to those reported for similar structures (Głowiak & Wnęk, 1985). The non-coordinated N atoms on opposite rings are in an inverted position with respect to each other as has been found in similar structures (Vreugdenhil, Birker, ten Hoedt, Verschoor & Reedijk, 1984; Głowiak & Wnęk, 1985). Three of the rings are close to being parallel with the linear I1—Cu—I2 direction [average torsion angle I2—Cu—N(coord.)—N(uncoord.) is −5° with a standard deviation of 1°]. The fourth has a significant tilt in the opposite direction [torsion angle I2—Cu—N3—N7 is 31 (1)°] (Fig. 3). The torsional twisting of one of the imidazole rings is unusual as most tetrakis(imidazole)copper(II) complexes have symmetries with a *trans* pair of imidazoles or a point symmetry center of inversion. In $[\mathrm{Cu(imH)_4SO_4}]$ and $[\mathrm{Cu(imH)_4\{P(C_6H_5O)_2O_2\}_2}]$ the maximum torsional angle of a *trans* pair of the imidazole rings with respect to the $\mathrm{CuN_4}$ equatorial plane is 30° (Fransson & Lundberg, 1972; Głowiak & Wnęk, 1985). In $[\mathrm{Cu(imH)_4(ClO_4)_2}]$ and $[\mathrm{Cu(imH)_4(NO_3)_2}]$ the maximum torsional angle of a *trans* pair of the imidazole rings is 19° (Ivarsson, 1973; McFadden,

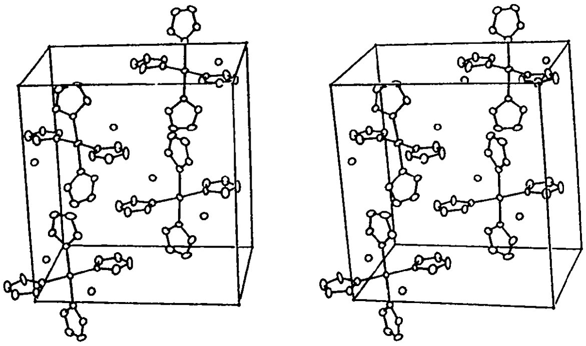

Fig. 1. Stereoscopic view of the unit cell looking down the *b* axis with the *c* axis horizontal and the *a* axis vertical.

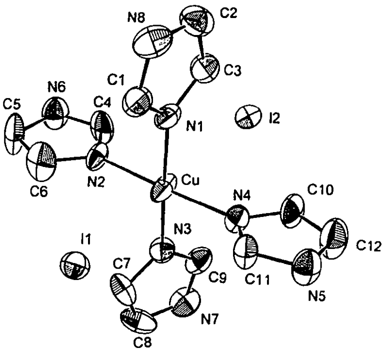

Fig. 2. Illustration of the structure of one formula unit of $[\mathrm{Cu(C_3H_4N_2)_4]I_2}$ and the atom-numbering scheme. H atoms have been omitted.

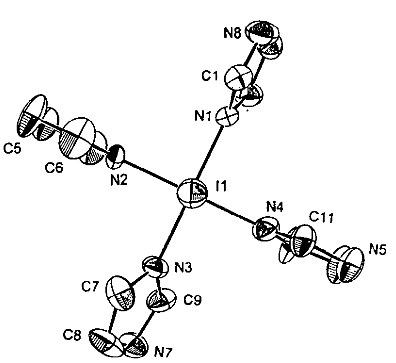

Fig. 3. View down the I1—Cu—I2 direction showing the extent of tipping of the imidazole rings from being parallel with the Cu—I bonds.

McPhail, Garner & Mabbs, 1976). The complex [$Cu(imH)_4(O_2CCH_2OCH_3)_2$] has a *trans* pair of imidazole rings with a torsional angle of 26° and another pair of imidazoles with a torsional angle of $-26°$ (Prout, Allison & Rossotti, 1976).

An unusual feature of this tetrakis(imidazole)-copper(II) complex is the unequal bond lengths for the two iodide ligands. The lack of improper rotations in the point symmetry signifies chirality and the absolute configuration of the chiral complex has been determined using the η parameter (Rogers, 1981) for the selected crystal.

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Acta Cryst. (1993). **C49**, 1424–1426

Structure of Bis[*cis*-1,2-bis(diphenylphosphino)ethylene]dichlororhenium(II) Hexane Solvate

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Abstract. [$ReCl_2(C_{26}H_{22}P_2)_2$].$C_6H_{14}$ (I), M_r = 1136.11, monoclinic, $P2_1/c$, a = 11.426 (2), b = 13.078 (2), c = 17.323 (3) Å, β = 95.27 (1)°, V = 2577.7 (6) Å^3, Z = 2, D_x = 1.46 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 26.5 cm^{-1}, $F(000)$ = 1150, T = 296 K, R = 0.034 for 3269 [$F_o^2 > 3\sigma(F_o^2)$] reflections. The Re atom in (I) is located on an inversion point, fixed at the origin. One Cl and a complete tertiary phosphine ligand ($Ph_2PCHCHPPh_2$) are situated around the Re atom so as to generate a pseudo-octahedral geometry with a P1—Re—P2 angle of 79.07 (5)° and P1—Re—P2′ angle of 100.93 (5)°. The Re—Cl, Re—P1 and Re—P2 distances are 2.432 (2), 2.401 (2) and 2.398 (2) Å, respectively. Bond dis-

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