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Tetra-μ_3-iodo-tetrakis[(cyclohexyldiphenylphosphine-κP)copper(I)]

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.011 Å
R factor = 0.061
wR factor = 0.125
Data-to-parameter ratio = 24.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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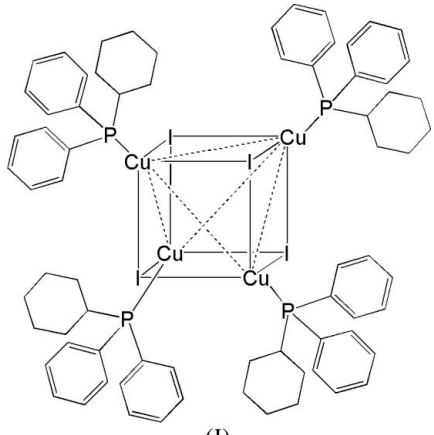
The molecule of the title compound, $[Cu_4I_4(C_{18}H_{21}P)_4]$, which lies on a crystallographic twofold rotation axis, displays a cubane-like Cu_4I_4 core.

Comment

Phosphine complexes of copper(I) halides $[(CuXL_n)_m]$ (X = halogen and L = phosphine), which display a wide range of coordination geometries, are useful as catalysts, as precursors to organocopper reagents (Taylor, 1994) and as starting materials for the preparation of heterometallic complexes (Albano *et al.*, 1995; Kudinov *et al.*, 1993). It was proposed that the title complex, (I), has an irregular cubane structure (Churchill & Kalra, 1974*a*,*b*,*c*; Churchill & Rotella, 1977), which is confirmed in the present study. The tetranuclear molecule lies on a crystallographic twofold rotation axis (Fig. 1).



(I)

The Cu_4I_4 core is a slightly irregular cubane with alternating copper(I) and iodide ions. The Cu atom exists in a tetrahedral environment, being linked to three I atoms and to the P atom of the cyclohexyldiphenylphosphine ligand.

The copper–iodine bond lengths show a significant range of values. The average of the bond lengths is in good agreement with the values of 2.6837 (13) and 2.6767 (15) Å in the Cu_4I_4 cores of other regular cubane-like adducts (Churchill & Kalra, 1974*a*,*b*,*c*; Churchill & Rotella, 1977). The six copper–copper contact distances are similar to reported values. The iodine–iodine contacts are also comparable to reported values, as are the copper–phosphorus bond distances.

There are van der Waals repulsive forces that may be responsible for the distortion of the six faces of the cubane core; the distortion manifests itself in the small Cu—I—Cu angles and in the non-planarity of the four-membered rings defining the faces of the cube.

Experimental

Cyclohexyldiphenylphosphine (0.235 g, 1 mmol) and copper(I) iodide (0.135 g, 2 mmol) were dissolved in 30 ml of acetone. After refluxing the mixture for one day, the hot solution was filtered. Cuboidal crystals were obtained upon recrystallization from the same solvent.

Crystal data

$[Cu_4I_4(C_{18}H_{21}P)_4]$
M_r = 1835.03
Monoclinic, $C2/c$
a = 22.7712 (3) Å
b = 15.6704 (3) Å
c = 21.9311 (5) Å
β = 116.054 (1)°
V = 7030.5 (2) Å^3
Z = 4
D_x = 1.734 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 3.08 mm^{-1}
T = 150 (2) K
Cuboid, colorless
0.26 × 0.22 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.501, T_{max} = 0.578
(expected range = 0.469–0.540)
63740 measured reflections
8032 independent reflections
6289 reflections with $I > 2\sigma(I)$
R_{int} = 0.127
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.125
S = 1.21
8032 reflections
331 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 97.862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.00 e Å^{-3}
$\Delta\rho_{min}$ = −0.92 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

I1—Cu1	2.6976 (11)	I2—Cu2^{i}	2.7667 (11)
I1—Cu1^{i}	2.6849 (10)	Cu1—Cu2	2.8631 (13)
I1—Cu2	2.6645 (10)	Cu1—Cu2^{i}	3.0292 (13)
I2—Cu2	2.6543 (11)	Cu2—Cu2^{i}	3.0570 (18)
I2—Cu1	2.7077 (10)		
Cu1^{i}—I1—Cu1	70.07 (4)	I2—Cu2—I1	115.10 (4)
Cu2—I1—Cu1^{i}	68.98 (3)	I2—Cu2—I2^{i}	104.49 (3)
Cu2—I1—Cu1	64.54 (3)	I1—Cu2—I2^{i}	106.78 (4)
Cu2—I2—Cu1	64.54 (3)	I2—Cu2—Cu1	58.64 (3)
Cu2—I2—Cu2^{i}	68.62 (3)	I1—Cu2—Cu1	58.29 (3)
Cu1—I2—Cu2^{i}	67.18 (3)	I2^{i}—Cu2—Cu1	107.27 (4)
I1^{i}—Cu1—I1	103.98 (3)	I2—Cu2—Cu1^{i}	105.64 (4)
I1^{i}—Cu1—I2	107.90 (3)	I1—Cu2—Cu1^{i}	55.83 (3)
I1—Cu1—I2	112.27 (4)	I2^{i}—Cu2—Cu1^{i}	55.48 (3)
I1^{i}—Cu1—Cu2	107.34 (4)	Cu1—Cu2—Cu1^{i}	63.18 (4)
I1—Cu1—Cu2	57.17 (3)	I2—Cu2—Cu2^{i}	57.43 (3)
I2—Cu1—Cu2	56.83 (3)	I1—Cu2—Cu2^{i}	102.56 (2)
I1^{i}—Cu1—Cu2^{i}	55.19 (3)	I2^{i}—Cu2—Cu2^{i}	53.95 (3)
I1—Cu1—Cu2^{i}	102.49 (4)	Cu1—Cu2—Cu2^{i}	61.45 (3)
I2—Cu1—Cu2^{i}	57.34 (3)	Cu1^{i}—Cu2—Cu2^{i}	56.12 (3)
Cu2—Cu1—Cu2^{i}	62.43 (4)		

Symmetry code: (i) $-x+1, y, -z+\frac{3}{2}$.

The data did not diffract well and the mosaicity was high so the R_{int} value was high. H atoms were placed in calculated positions (C—H = **0.95–1.00** Å) and included as riding atoms, with U_{iso}(H) values of $1.2U_{eq}$ of the attached C atoms. The final difference Fourier map had a large peak/hole 1.00 Å from atom C14..

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

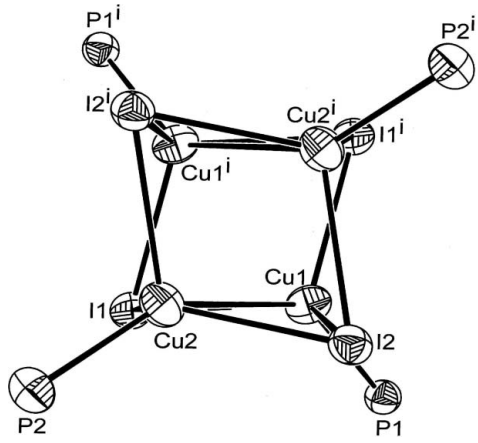


Figure 1
The central coren of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The symmetry code is given in Table 1.

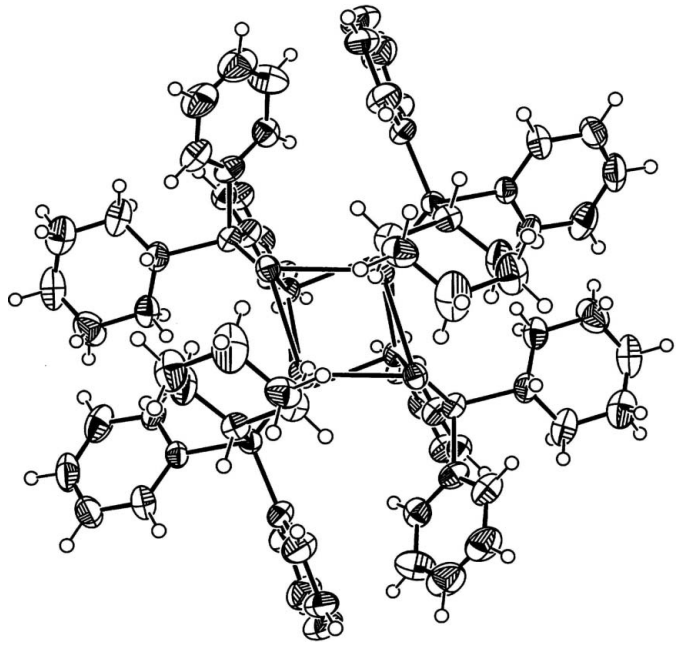


Figure 2
The molecular structure of complex (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

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