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## Key indicators

Single-crystal X-ray study

T = 150 K

Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$ 

R factor = 0.031

wR factor = 0.075

Data-to-parameter ratio = 20.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Di- $\mu$ -iodo-1 $\kappa^1$ :2 $\kappa^1$ -tris(tri-*m*-tolylphosphine)-1 $\kappa^2$ P,P':2 $\kappa^2$ P''-dicopper(I): a new polymorphThe structure of the title compound,  $[\text{Cu}_2\text{I}_2(\text{C}_{21}\text{H}_{21}\text{P})_3]$ , was redetermined at low temperature (150 K) and shown to be a new triclinic polymorph.

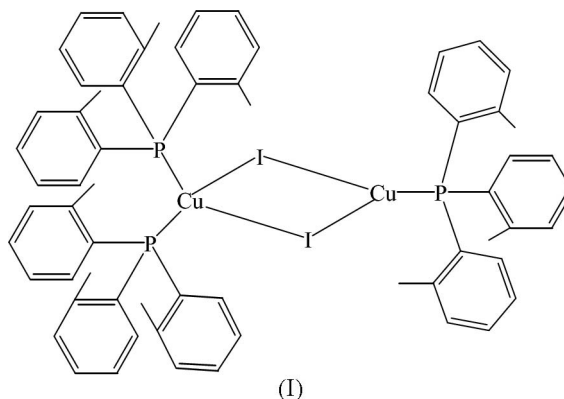
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## Comment

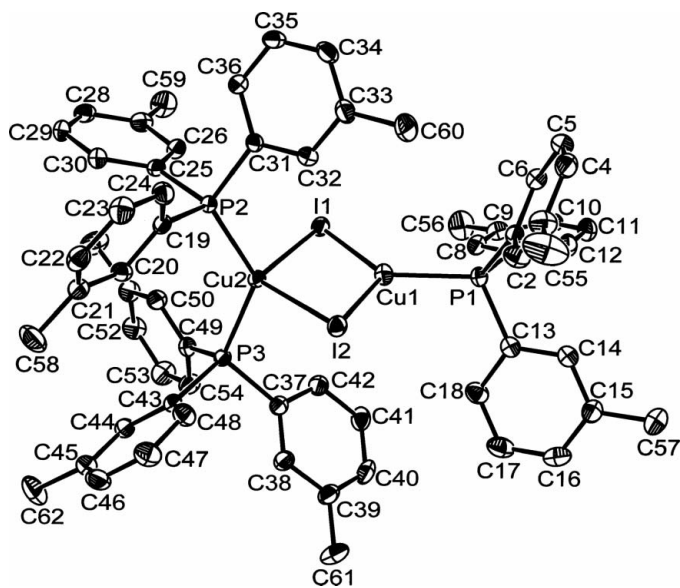
The title compound, (I), is a binuclear copper(I) complex, in which the Cu atoms are bridged by two I atoms. A total of three molecules of tri-*m*-tolylphosphine are coordinated to the two copper centres. The structure of the same compound was reported by Akrivos *et al.* (1993) and shown also to be triclinic, space group  $P\bar{1}$ . This polymorph, (II), has a significantly longer *c* axis [24.635 Å compared to 19.0630 (3) Å for the present structure, (I)] and slightly smaller cell angles, considering the reduced cells of both polymorphs.



The molecular structure of (I) is illustrated in Fig. 1 and selected bond distances and angles are given in Table 1. In (I), atom Cu1 is coordinated by one P atom (P1), and atom Cu2 is coordinated by two P atoms (P2 and P3). The copper centres are bridged by two I atoms. The intramolecular Cu...Cu distance of 2.9551 (4) Å is decreased by *ca* 0.05 Å compared with that in (II). An interesting difference between the two polymorphs is that the Cu—I distances are inverted. That is, in (I), the Cu1—I distances average 2.5544 (1) Å and the Cu2—I distances average 2.750 (1) Å. This is exactly the opposite situation in polymorph (II) where the corresponding average distances are 2.767 and 2.549 Å, respectively. The Cu—I—Cu angles are very similar. The dihedral angle between the Cu1/I1/I2 and Cu2/I1/I2 planes of 3.75 (12)° is smaller than the value of 8.56° in polymorph (II).

## Experimental

A suspension of tri-*m*-tolylphosphine (0.310 g, 1 mmol) and copper(I) iodide (0.382 g, 2 mmol) in 2-propanol (50 ml) was refluxed



**Figure 1**  
View of the molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

for 16 h. The resulting solution was filtered while hot. Slow evaporation of the solvent at room temperature gave colourless crystals of (I).

**Crystal data**

[Cu<sub>2</sub>I<sub>2</sub>(C<sub>21</sub>H<sub>21</sub>P)<sub>3</sub>]  
*M<sub>r</sub>* = 1293.92  
 Triclinic, *P* $\bar{1}$   
*a* = 11.6770 (1) Å  
*b* = 13.5461 (1) Å  
*c* = 19.0630 (3) Å  
 $\alpha$  = 86.0220 (5)°  
 $\beta$  = 86.1216 (5)°  
 $\gamma$  = 72.6081 (5)°  
*V* = 2867.07 (6) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.499 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 12975 reflections  
 $\theta$  = 2.9–27.5°  
 $\mu$  = 1.94 mm<sup>-1</sup>  
*T* = 150 (2) K  
 Prism, colourless  
 0.20 × 0.15 × 0.15 mm

**Data collection**

Nonius KappaCCD diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (Blessing, 1995)  
*T<sub>min</sub>* = 0.690, *T<sub>max</sub>* = 0.749  
 40258 measured reflections  
 12975 independent reflections  
 11475 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.045  
 $\theta_{max}$  = 27.6°  
*h* = -15 → 15  
*k* = -17 → 17  
*l* = -24 → 24

**Refinement**

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.031  
*wR*(*F*<sup>2</sup>) = 0.075  
*S* = 1.03  
 12975 reflections  
 640 parameters  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0251P)^2 + 3.1211P]$   
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 ( $\Delta/\sigma$ )<sub>max</sub> = 0.002  
 $\Delta\rho_{max}$  = 0.70 e Å<sup>-3</sup>  
 $\Delta\rho_{min}$  = -1.18 e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Cu1...Cu2	2.9551 (4)	Cu1–I2	2.5481 (4)
Cu1–P1	2.2298 (7)	Cu2–P3	2.2737 (7)
Cu2–P2	2.2687 (7)	Cu2–I2	2.7454 (3)
Cu1–I1	2.5403 (3)	Cu2–I1	2.7548 (3)
P1–Cu1–I1	126.33 (2)	P2–Cu2–I1	102.441 (19)
P1–Cu1–I2	114.78 (2)	P3–Cu2–I1	106.48 (2)
I1–Cu1–I2	118.859 (12)	I2–Cu2–I1	105.603 (10)
P2–Cu2–P3	130.66 (2)	Cu1–I1–Cu2	67.706 (10)
P2–Cu2–I2	109.470 (19)	Cu1–I2–Cu2	67.750 (10)
P3–Cu2–I2	100.138 (19)		

The H atoms were placed in calculated positions (aromatic C–H = 0.95 Å and methyl C–H = 0.98 Å), and refined using a riding model, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(aromatic C) and 1.5*U*<sub>eq</sub>(methyl C). The deepest electron-density hole lies 0.86 Å from atom I2.

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, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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