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The absolute structure of tetrakis(trimethylphosphine)copper(I) dichlorocopper(I)

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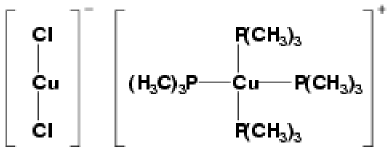
The title compound, $[\mathrm{Cu(C_3H_9P_4)_4}][\mathrm{CuCl_2}]$, previously reported by Chi *et al.* [*J. Chem. Soc. Dalton Trans.* (1992), pp. 3111–3117] has been rerefined with new intensity data. Geometric parameters agree quite well. However, our results are of significantly higher precision. Furthermore, our data indicate a different absolute structure than the one reported by Chi *et al.* The Cu, Cl atoms and one of the P atoms are located on a threefold rotation axis. As a result, there is just one third of both ions in the asymmetric unit.

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(P–C) = 0.004 Å
R factor = 0.031
wR factor = 0.080
Data-to-parameter ratio = 35.6

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



$[\mathrm{Cl{-}Cu{-}Cl}]^{-}\,[\mathrm{Cu(P(CH_3)_3)_4}]^{+}$

(I)

Comment

Recently, we reported the X-ray crystal structure analyses of $[\mathrm{Cu(NH_3)Cl}]$ and $[\mathrm{Cu(NH_3)_2}]\mathrm{Br}$ (Margraf *et al.*, 2003). In the solid state, both compounds feature linear two coordinated copper(I) fragments with short copper(I)–copper(I) contacts. Now, we are interested in the syntheses and solid-state characterizations of new $\mathrm{Cu^I}$ complexes with sterically more demanding ligands, such as $^t\mathrm{Bu_3SiPH_2}$ and $^t\mathrm{Bu_2PhSiPH_2}$. Therefore, we have prepared the title compound, (I), as a starting material.

The structure of (I) was previously reported by Chi *et al.* (1992). The geometric parameters of both determinations agree quite well, but the present work is of significantly improved precision. Furthermore, our data indicate that the absolute structure is different from that reported by Chi *et al.* (1992). A perspective view of (I) is shown in Fig. 1. The Cu and Cl atoms and one of the P atoms are located on a threefold rotation axis. As a result, there is just one third of both ions in the asymmetric unit.

The different absolute structure of the title compound does not show that the previous result was wrong; presumably there is spontaneous resolution on crystallization, and the choice of a particular handedness for the crystal studied is arbitrary. This might demonstrate that an achiral compound can crystallize in both enantiomers.

Experimental

The title compound was synthesized by dropwise addition of $\mathrm{P(CH_3)_3}$ (152 mg, 2 mmol) to CuCl (99 mg, 1 mmol) in THF. Colourless single crystals were obtained by storing the THF solution at ambient temperature.

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Crystal data

$[Cu(C_3H_9P_4)_4][CuCl_2]$
$M_r = 502.27$
Cubic, $P2_13$
$a = 13.4832\ (11)$ Å
$V = 2451.2\ (3)$ Å^3
$Z = 4$
$D_x = 1.361$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5949 reflections
$\theta = 3.8$–$31.3°$
$\mu = 2.21$ mm^{-1}
$T = 173\ (2)$ K
Block, colourless
0.50 × 0.40 × 0.30 mm

Data collection

Stoe IPDS–II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
$T_{\min} = 0.380$, $T_{\max} = 0.521$
4186 measured reflections
2172 independent reflections
2035 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 31.3°$
$h = -18 \rightarrow 18$
$k = -11 \rightarrow 19$
$l = -18 \rightarrow 4$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.080$
$S = 1.05$
2172 reflections
61 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}
Absolute structure: Flack (1983), 686 Friedel pairs
Flack parameter = −0.012 (14)

All H atoms were located in a difference Fourier synthesis. They were refined with fixed individual displacement parameters [$U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$] using a riding model with C—H$_{\rm methyl}$ = 0.98 Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 1990).

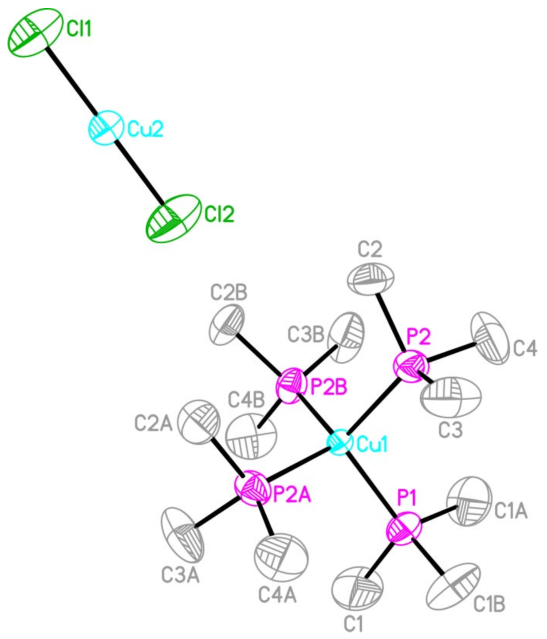

Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. Atoms marked with suffixes A and B are related to those with no suffixes by the symmetry codes (z, x, y) and (y, z, x), respectively. H atoms have been omitted.

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