metal-organic papers

Feng-Bo Xu,* Hu Xu,† Xue-Bing Leng, Hai-Bin Song, Qing-Shan Li, Ru-Yi Zou and Zheng-Zhi Zhang

State Key Laboratory of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China

† Additional correspondence author, email: xuhuz@eyou.com.

Correspondence e-mail: xfbo@eyou.com

{μ-9,10-Bis[(n-propylamino)methyl]-anthracene-κ²N:N'}bis{bis[6-chloro-2-(diphenylphosphino)pyridine-κP]-copper(I)} diperchlorate

The title compound, $[Cu_2(C_{15}H_{13}ClP)_4(C_{30}H_{28}N_2)](ClO_4)_2$, was synthesized by the reaction of 9,10-bis[(n-propylamino)-methyl]anthracene and 6-chloro-2-(diphenylphosphino)-pyridine with $[Cu(CH_3CN)_4]ClO_4$. The Cu atoms adopt a trigonal–planar geometry and the cation is centrosymmetric. Three-layer π–π stacking interactions are observed between the anthracene and pyridyl rings.

Comment

Metal complexes containing P,N-ligands have been subjected to intensive study for many years (Newkome, 1993; Zhang & Cheng, 1996; Espinet & Soulantica, 1999). The interest mainly stems from: (i) the versatile coordination abilities of P or N atoms in forming a growing number of mono- and polynuclear complexes (Schutte et al., 1997; Xu et al., 2002; Kuang et al., 1998); (ii) potential molecule-based chemosensors (Rogers & Wolf, 2002); (iii) molecular recognition (Xu et al., 2000); (iv) catalytic activity in some reactions (Drent et al., 1993; Loiseleur et al., 1999; Trost & Van Vranken, 1996). We have successfully designed and prepared the title dinuclear complex, (I), by self-assembly of P,N-ligands with Cu^{I}.

(I)
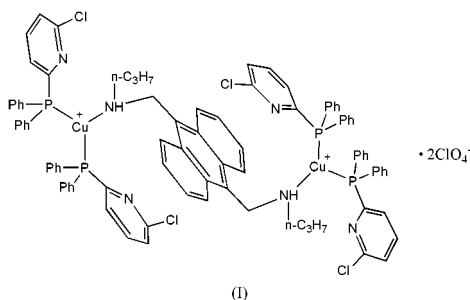
As shown in Fig. 1, each Cu^{I} atom adopts a trigonal–planar geometry, coordinated by one N and two P atoms. The cation is centrosymmetric. The N—Cu—P1, N—Cu—P2 and P1—Cu—P2 angles are 117.91 (15), 116.44 (15) and 125.13 (5)°, respectively. There is an intramolecular interaction involving three-layer π–π stacking interactions between the anthracene and two pyridyl rings. The anthracene and pyridyl rings are almost parallel, with dihedral angles of 9.7 (2)° and a separation distance of 3.771 (4) Å between two neighbouring rings.

Experimental

The synthesis of the title compound was carried out under an N_2 atmosphere. To a CH_2Cl_2 solution (20 ml) of 9,10-bis[(n-propyl-amino)methyl]anthracene (0.08 g, 0.25 mmol) and 6-chloro-2-(di-
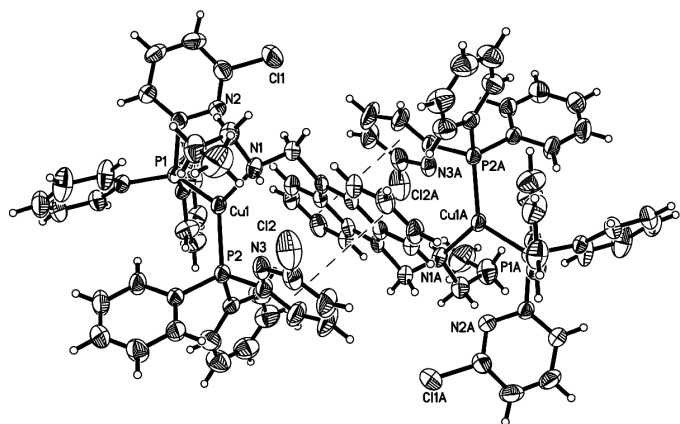
Figure 1
A view of the complex cation, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular π–π interaction is shown as a dashed line.

phenylphosphino)pyridine (0.279 g, 1 mmol) was added [Cu(CH₃CN)₄]ClO₄ (0.164 g, 0.5 mmol). The mixture was stirred for 4 h at room temperature. The mixture was then filtered and the filtrate concentrated to a small volume. Diethyl ether was added to give a white solid (0.29 g). Crystals were obtained by diffusion of diethyl ether into a clear solution of the crude product in CH_2Cl_2.

Crystal data

$[Cu_2(C_{15}H_{13}ClP)_4(C_{30}H_{28}N_2)]$- $(ClO_4)_2$	$D_x = 1.376$ Mg m^{-3}
$M_r = 1837.28$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 873 reflections
$a = 13.507$ (3) Å	$\theta = 2.5–23.5°$
$b = 18.359$ (4) Å	$\mu = 0.79$ mm^{-1}
$c = 17.968$ (4) Å	$T = 298$ (2) K
$\beta = 95.409$ (5)°	Block, colourless
$V = 4435.7$ (15) Å^3	$0.35 \times 0.25 \times 0.20$ mm
$Z = 2$	

Data collection

Bruker SMART CCD area-detector diffractometer	7801 independent reflections
φ and ω scans	3755 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.084$
	$\theta_{max} = 25.0°$
$T_{min} = 0.797$, $T_{max} = 0.858$	$h = -16 \rightarrow 14$
18343 measured reflections	$k = -21 \rightarrow 20$
	$l = -18 \rightarrow 21$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.059$	
$wR(F^2) = 0.136$	$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
7801 reflections	$(\Delta/\sigma)_{max} = 0.002$
578 parameters	$\Delta\rho_{max} = 0.73$ e Å^{-3}
	$\Delta\rho_{min} = -0.32$ e Å^{-3}

All H atoms, except the amine H atoms, were positioned geometrically with C—H distances of 0.98 or 0.93 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ or $1.2U_{eq}(C)$. The positions of the amine H atoms were initially located in a difference Fourier map and were refined with isotropic displacement parameters. The ClO_4^- anion and propyl chain are disordered over two positions each. The Cl—O and C—C bonds were restrained to 1.45 and 1.54 Å, respectively, and the O—Cl—O and C—C—C angles were restrained to 109.5°.
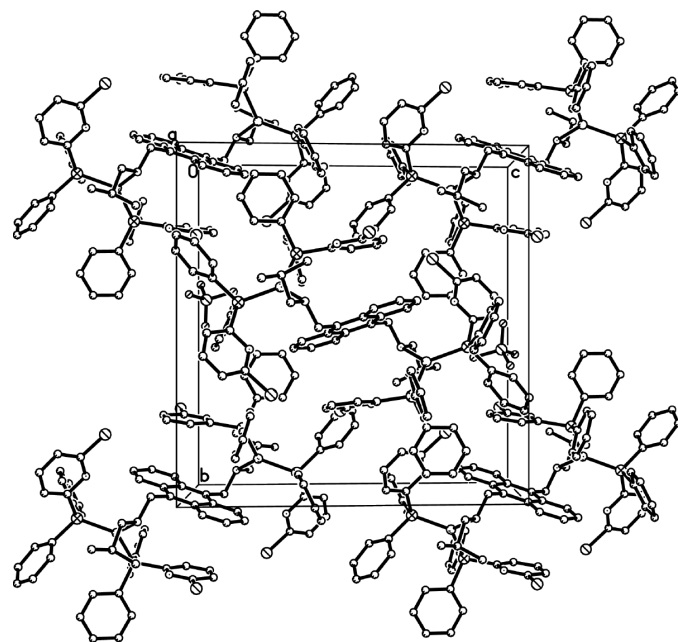

Figure 2
A view of the packing arrangement in the unit cell of (I). H atoms have been omitted for clarity.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998) and *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Bruker (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SMART* 1000 *Operation Manual* and *SAINT* (Version 5.00). Bruker AXS Inc., Madison, Wisconsin, USA.
Drent, E., Arnoldy, P. & Budzelaar, P. H. M. (1993). *J. Organomet. Chem.* **455**, 247–253.
Espinet, P. & Soulantica, K. (1999). *Coord. Chem. Rev.* **195**, 499–556.
Kuang, S. M., Zhang, Z. Z., Wang, Q. G. & Mak, T. C. W. (1998). *Chem. Commun.* pp. 581–582.
Loiseleur, O., Hayashi, M., Keenan, M., Schmees, N. & Pfaltz, A. (1999). *J. Organomet. Chem.* **576**, 16–22.
Newkome, G. R. (1993). *Chem. Rev.* **93**, 2067–2089.
Rogers, C. W. & Wolf, M. O. (2002). *Coord. Chem. Rev.* **233**, 341–350.
Schutte, R. P., Rettig, J., Joshi, A. M. & James, B. R. (1997). *Inorg. Chem.* **36**, 5809–5817.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELX*97 (includes *SHELXS*97, *SHELXL*97, *CIFTAB*). Release 97-2. University of Göttingen, Germany.
Trost, B. M. & Van Vranken, D. L. (1996). *Chem. Rev.* **96**, 395–422.
Xu, F. B., Li, Q. S., Zeng, X. S., Leng, X. B. & Zhang, Z. Z. (2002). *Organometallics*, **21**, 4894–4896.
Xu, F. B., Weng, L. H., Sun, L. J., Zhang, Z. Z. & Zhou, Z. F. (2000). *Organometallics*, **19**, 2658–2660.
Zhang, Z. Z. & Cheng, H. (1996). *Coord. Chem. Rev.* **147**, 1–39.