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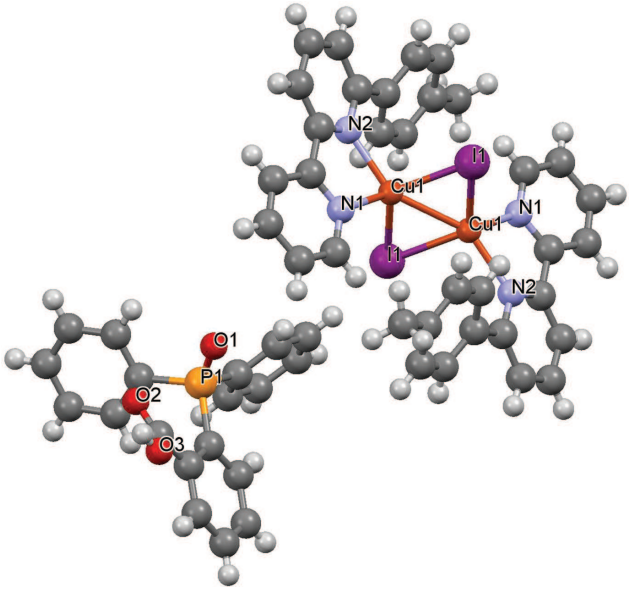
Crystal structure of di-μ-iodido-bis(6-(*p*-tolyl)-2,2′-bipyridine-κ²*N*,*N*′)dicopper(I) — 2-(diphenylphosphoryl)benzoic acid (1/2), $C_{36}H_{29}CuIN_2O_3P$



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Abstract

$C_{36}H_{29}CuIN_2O_3P$, triclinic, $P\bar{1}$ (no. 2), $a = 9.4713(3)$ Å, $b = 11.8586(4)$ Å, $c = 14.7597(5)$ Å, $\alpha = 93.5250(10)°$, $\beta = 90.61(10)°$, $\gamma = 108.99(10)°$, $V = 1563.71(9)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0279$, $wR_{ref}(F^2) = 0.0688$, $T = 293$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow single crystals
Size:	0.25 × 0.2 × 0.1 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.78 mm^{-1}
Diffractometer, scan mode:	CCD area detector, ϕ and ω scans
θ_{max}:	28.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11899, 6810, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5193
$N(param)_{refined}$:	398
Programs:	Bruker *SMART*, *SAINT* [4], SHELX [5]

Source of material

6-(4-methanylphenyl)-2,2′-bipyridine was prepared by a literature method [3]. CuI and 2-(diphenylphosphinyl)-benzoic were commercial available. Equivalent amount (1 mmol) of CuI, 6-(4-methanylphenyl)-2,2′-bipyridine and 2-(diphenylphosphinyl)benzoic acid were added to 30 mL dichloromethane under a nitrogen atmosphere. The mixture was reacted for 2 h at room temperature. Then the solvent was removed. Yellow crystals were obtained from the solution of dichloromethane by vapor diffusion with diethyl ether.

Experimenal details

H atoms on C atoms were positioned geometrically refined using a riding model with C—H = 0.93–0.96 Å, and $U_{iso}(H) = 1.2\ U_{eq}(C)$ or $1.5\ U_{eq}(C)$. The hydrogen atom in the carboxyl group was fixed to OH distances of 0.82(1) Å [$U_{iso}(H) = 1.2U_{eq}(O)$].

Comment

Copper(I) complexes with bipyridine ligands have attracted much attention for their low-lying charge-transfer (CT) excited states, and potential applications in electrochemical devices and in solar energy conversion [1]. In addition, 2-(diphenylphosphinyl)benzoate is also a useful ligand in transitional metal complexes, and has been used for the

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
I1	0.202358(17)	0.149018(15)	0.455673(11)	0.04863(7)
Cu1	−0.05539(3)	−0.01061(3)	0.420358(18)	0.04417(9)
P1	0.62328(7)	0.27130(6)	0.13007(4)	0.03666(14)
O1	0.47814(19)	0.17238(15)	0.13022(11)	0.0460(4)
N2	−0.2275(2)	0.01138(16)	0.33889(12)	0.0347(4)
O2	0.6287(2)	0.11778(16)	−0.03034(11)	0.0560(5)
O3	0.7194(2)	−0.03213(16)	−0.01650(12)	0.0545(5)
H3	0.6585	−0.0644	−0.0579	0.082*
N1	−0.0349(2)	−0.10711(18)	0.30224(13)	0.0412(5)
C18	0.7908(3)	0.2271(2)	0.14072(14)	0.0354(5)
C25	0.6319(3)	0.3769(2)	0.22558(14)	0.0359(5)
C31	0.6443(3)	0.3567(2)	0.03063(15)	0.0403(5)
C11	−0.2880(2)	0.1557(2)	0.44203(15)	0.0387(5)
C1	0.0744(3)	−0.1524(2)	0.28324(18)	0.0517(7)
H1B	0.1486	−0.1437	0.3274	0.062*
C6	−0.2591(3)	−0.0655(2)	0.26424(14)	0.0378(5)
C16	−0.1443(3)	0.2361(2)	0.45245(16)	0.0440(6)
H16A	−0.0708	0.2276	0.4140	0.053*
C35	0.5254(4)	0.4111(3)	−0.09626(19)	0.0656(9)
H35A	0.4396	0.4027	−0.1309	0.079*
C5	−0.1417(3)	−0.1183(2)	0.23842(15)	0.0386(5)
C7	−0.3934(3)	−0.0929(2)	0.21540(16)	0.0491(6)
H7A	−0.4120	−0.1445	0.1634	0.059*
C13	−0.3559(3)	0.2627(3)	0.56904(19)	0.0597(8)
H13A	−0.4283	0.2707	0.6087	0.072*
C12	−0.3940(3)	0.1698(3)	0.50281(18)	0.0505(6)
H12A	−0.4910	0.1163	0.4986	0.061*
C29	0.7384(3)	0.5672(2)	0.30983(19)	0.0551(7)
H29A	0.8092	0.6432	0.3157	0.066*
C20	0.9505(3)	0.1135(2)	0.09429(16)	0.0453(6)
H20A	0.9689	0.0558	0.0550	0.054*
C19	0.8201(3)	0.1405(2)	0.08222(14)	0.0374(5)
C15	−0.1085(3)	0.3287(2)	0.51909(18)	0.0528(7)
H15A	−0.0113	0.3818	0.5242	0.063*
C26	0.5284(3)	0.3423(2)	0.29182(17)	0.0505(6)
H26A	0.4569	0.2666	0.2864	0.061*
C36	0.5160(3)	0.3468(2)	−0.01990(17)	0.0519(7)
H36A	0.4237	0.2971	−0.0026	0.062*
C22	1.0239(3)	0.2523(2)	0.22270(16)	0.0457(6)
H22A	1.0910	0.2892	0.2705	0.055*
C14	−0.2132(3)	0.3443(3)	0.57815(18)	0.0556(7)
C28	0.6355(4)	0.5309(3)	0.37571(19)	0.0671(8)
H28A	0.6372	0.5817	0.4266	0.081*
C9	−0.4674(3)	0.0344(2)	0.31997(17)	0.0475(6)
H9A	−0.5383	0.0679	0.3406	0.057*
C24	0.7113(3)	0.0750(2)	0.00626(15)	0.0414(6)
C10	−0.3291(3)	0.0631(2)	0.36593(15)	0.0375(5)
C30	0.7373(3)	0.4917(2)	0.23515(16)	0.0450(6)
H30A	0.8071	0.5171	0.1906	0.054*
C23	0.8939(3)	0.2806(2)	0.21178(15)	0.0411(6)
H23A	0.8750	0.3364	0.2527	0.049*
C4	−0.1386(3)	−0.1747(2)	0.15345(16)	0.0507(6)
H4A	−0.2117	−0.1806	0.1092	0.061*
C2	0.0817(3)	−0.2113(3)	0.2012(2)	0.0602(8)
H2A	0.1580	−0.2432	0.1904	0.072*
C21	1.0538(3)	0.1700(2)	0.16316(17)	0.0475(6)
H21A	1.1422	0.1526	0.1691	0.057*
C32	0.7788(3)	0.4311(3)	0.00403(18)	0.0623(8)
H32A	0.8658	0.4374	0.0367	0.075*
C8	−0.4976(3)	−0.0436(3)	0.24429(17)	0.0520(7)
H8A	−0.5888	−0.0627	0.2128	0.062*
C3	−0.0265(3)	−0.2218(3)	0.13566(19)	0.0601(8)
H3A	−0.0238	−0.2606	0.0793	0.072*
C34	0.6582(4)	0.4857(3)	−0.12060(19)	0.0698(9)
H34A	0.6630	0.5297	−0.1712	0.084*
C17	−0.1736(4)	0.4480(3)	0.6487(2)	0.0895(11)
H17A	−0.2298	0.4242	0.7021	0.134*
H17B	−0.1970	0.5136	0.6250	0.134*
H17C	−0.0687	0.4723	0.6641	0.134*
C33	0.7861(4)	0.4972(3)	−0.0712(2)	0.0811(11)
H33A	0.8775	0.5490	−0.0881	0.097*
C27	0.5299(4)	0.4192(3)	0.3663(2)	0.0734(9)
H27A	0.4588	0.3952	0.4104	0.088*

construction of coordination polymers [2]. We intended to synthesize a polynuclear copper(I) complex using two different ligands. But unexpectely, the title complex was obtained.

The asymmetric unit of the title crystal structure contains the coordinated 2-(diphenylphosphoryl)benzoic acid and one half of a $(C_{17}H_{14}N_2)_2Cu(\mu\text{-}I)_2$ complex. Each copper(I) adopts a distorted tetrahedral coordination (neglection of the Cu-Cu interaction), with the N1—Cu1—N2 and I1—Cu1—I1A angles of 79.80(7) and 121.94(1)°, respectively. The Cu—N and Cu—I distances are within the normal ranges [3]. In addition, a Cu-Cu interaction was found in the title compound, with the Cu1—Cu1A distance of 2.5287(5) Å. Owing to the chelate effect, the two pyridine ring moieties are almost coplanar [dihedral angle of 16.5(1)°], while the pendant aromatic ring is twist with the adjacent pyridine fragment [dihedral angle of 37.7(1)°].

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