Crystal structure of bis(triphenylphospine-κP)bis(μ-chloro-κCl) bis(isoquinoline-κN)copper(I), $[CuCl(PC_{18}H_{15})(i\text{-}C_9H_7N)]_2$

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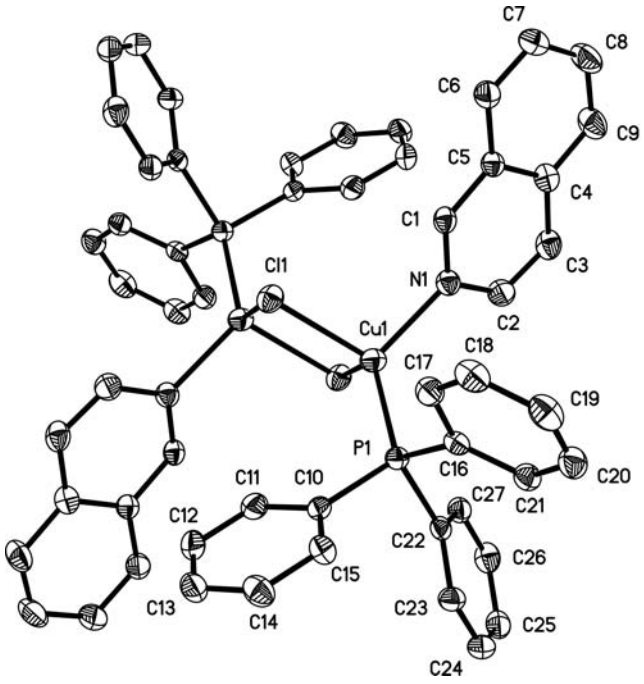

Abstract

$C_{54}H_{44}Cl_2Cu_2N_2P_2$, monoclinic, $C12/c1$ (no. 15), a = 27.870(2) Å, b = 9.906(1) Å, c = 18.595(2) Å, β = 115.719(2)°, V = 4625.2 Å^3, Z = 4, $R_{gt(F)}$ = 0.032, $wR_{ref(F^2)}$ = 0.078, T = 298 K.

Source of material

The title compound was obtained by the reaction of CuCl and PPh_3 (molar ratio 1:1) in the presence of isoquinoline (1 ml) in the mixed solution of CH_3OH (5 ml) and CH_2Cl_2 (5 ml) for 4 hours at ambient temperature. The insoluble residues were removed by filtration, and the filtrate was evaporated slowly at room temperature for about one week to yield yellow crystals. Crystals suitable for single crystal X-ray diffraction investigation were extracted directly from the sample as prepared.

Discussion

Copper(I) complexes containing triphenylphosphine (PPh_3) and heterocyclic nitrigen ligands have been studied well because of their interesting coordination chemistry and potential applications in photography, electrochemical processes, antimicrobial and antitumor activities [1-3]. In the course of our work on the syntheses of Cu(I) compounds, we obtained a series of complexes containing phosphorus and nitrogen ligands [2,5-7]. The complexes of of quinoline(C_9H_7N) and/or isoquinoline (i-C_9H_7N) $[CuX(PPh_3)(C_9H_7N]_2$ (X = Br for **2**, X = I for **3**) [5,6], $[Cu(CH_2(P(C_6H_5)_2)_2)(C_9H_7N)(i\text{-}C_9H_7N)]_2I_2$ (**4**) [7], $[Cu_2(CH_2(P(C_6H_5)_2)_2)_2(i\text{-}C_9H_7N)]Br_2 \cdot C_{10}H_8 \cdot C_9H_7N \cdot CH_3CN$ (**5**) [8] have been reported.

The crystal structure of the title compound consists of inversion-symmetric dimers with a diamond-shaped Cu_2Cl_2 group at the center. Each Cu atom is coordinated by one P atom from PPh_3, one N atom from isoquinoline and two bridged chloride ions. The complexes $[CuBr(PPh_3)(C_9H_7N]_2$ (**2**) [5], $[CuI(PPh_3)(C_9H_7N)]_2$ (**3**) [6] have a similar crystal structure to the title complex. The Cu1—Cl1 (2.3840(7) - 2.4189(8) Å), Cu1—P1 (2.1945(8) Å) and Cu1—N1 (2.066(2) Å) bond lengthes in the title crystal structure are shorter than the corresponding distances in (**2**) and (**3**) [in (**2**), Cu—Br (2.520(1) - 2.573(1) Å); Cu—P (2.216(1) Å); in (**3**), Cu—I (2.6617(6) - 2.7059(5) Å), Cu—P (2.247(1) Å) and Cu—N (2.097(3) Å)]. The different distances in these complexes may be due to the difference of the anions. The two N–Cu–P bond angles (120.23(7)°) are larger than the N–Cu–P bond angles (117.20(8)°) in compound (**3**) and also much larger than those in complex (**2**) (95.83(2)° and 118.1(1)°). While the bond angle N–Cu–Cl (104.11(6)°) and the bond angle P–Cu–Cl (111.67(3)°) are shorter than the corresponding bond angle N–Cu–I (106.25(8)°) and bond angle P–Cu–I (114.70(3)°) in (**3**).

The result of this research gives an important information for designing and synthesizing new compounds containing N- and P-ligands. From the title complexes (**4**) and (**5**), we can see that isoquninoline has strong coordination ability to Cu(I) atom. In the reaction of preparing complexes (**4**) and (**5**), the copper atom is coordinated by isoquinoline even if only small amount of isoquinoline is added as impurity of quinoline.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.25 × 0.33 × 0.48 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.44 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11301, 4068
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2986
$N(param)_{refined}$:	280
Programs:	SHELXS-97, SHELXL-97 [9]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*f*	0.5310	0.3523	0.6870	0.044
H(2)	8*f*	0.4479	0.1306	0.5019	0.057
H(3)	8*f*	0.4810	–0.0616	0.5698	0.058
H(6)	8*f*	0.5931	0.2484	0.8151	0.055
H(7)	8*f*	0.6314	0.0589	0.8867	0.070
H(8)	8*f*	0.6079	–0.1526	0.8280	0.076
H(9)	8*f*	0.5462	–0.1762	0.6996	0.069
H(11)	8*f*	0.4115	0.7083	0.4199	0.053
H(12)	8*f*	0.3929	0.9349	0.3909	0.066
H(13)	8*f*	0.3309	1.0414	0.4209	0.064
H(14)	8*f*	0.2866	0.9244	0.4806	0.062
H(15)	8*f*	0.3048	0.6977	0.5101	0.051
H(17)	8*f*	0.4145	0.5782	0.6458	0.052
H(18)	8*f*	0.3905	0.5595	0.7491	0.060
H(19)	8*f*	0.3170	0.4336	0.7339	0.065
H(20)	8*f*	0.2681	0.3239	0.6153	0.060
H(21)	8*f*	0.2896	0.3475	0.5095	0.049
H(23)	8*f*	0.2593	0.5304	0.3823	0.049
H(24)	8*f*	0.1998	0.4296	0.2658	0.059
H(25)	8*f*	0.2277	0.2619	0.2072	0.059
H(26)	8*f*	0.3151	0.1910	0.2657	0.058
H(27)	8*f*	0.3750	0.2898	0.3830	0.048

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	8*f*	0.45695(1)	0.43332(3)	0.51918(2)	0.0348(2)	0.0429(2)	0.0403(2)	0.0026(2)	0.0130(2)	0.0017(2)
Cl(1)	8*f*	0.51935(3)	0.60575(7)	0.58976(4)	0.0461(4)	0.0462(4)	0.0355(4)	–0.0085(3)	0.0173(3)	–0.0088(3)
P(1)	8*f*	0.37472(3)	0.49779(7)	0.48616(4)	0.0314(4)	0.0325(4)	0.0313(4)	0.0003(3)	0.0117(3)	0.0004(3)
N(1)	8*f*	0.48688(9)	0.2625(2)	0.5880(1)	0.042(1)	0.037(1)	0.037(1)	–0.002(1)	0.018(1)	0.000(1)
C(1)	8*f*	0.5216(1)	0.2677(3)	0.6634(2)	0.044(2)	0.031(1)	0.038(2)	–0.005(1)	0.019(1)	–0.003(1)
C(2)	8*f*	0.4726(1)	0.1366(3)	0.5550(2)	0.053(2)	0.046(2)	0.036(2)	–0.005(2)	0.013(1)	–0.005(1)
C(3)	8*f*	0.4925(1)	0.0211(3)	0.5952(2)	0.062(2)	0.037(2)	0.046(2)	–0.009(1)	0.023(2)	–0.010(1)
C(4)	8*f*	0.5307(1)	0.0248(3)	0.6757(2)	0.051(2)	0.038(2)	0.041(2)	–0.000(1)	0.026(2)	–0.003(1)
C(5)	8*f*	0.5456(1)	0.1539(3)	0.7109(2)	0.038(2)	0.037(2)	0.034(2)	–0.001(1)	0.020(1)	0.001(1)
C(6)	8*f*	0.5836(1)	0.1639(3)	0.7912(2)	0.050(2)	0.043(2)	0.042(2)	–0.004(1)	0.017(2)	0.001(1)
C(7)	8*f*	0.6063(1)	0.0514(3)	0.8338(2)	0.058(2)	0.058(2)	0.046(2)	0.001(2)	0.011(2)	0.011(2)
C(8)	8*f*	0.5919(1)	–0.0762(3)	0.7983(2)	0.073(2)	0.045(2)	0.064(2)	0.017(2)	0.022(2)	0.018(2)
C(9)	8*f*	0.5552(1)	–0.0905(3)	0.7218(2)	0.078(2)	0.033(2)	0.058(2)	0.006(2)	0.027(2)	0.001(2)
C(10)	8*f*	0.3595(1)	0.6784(2)	0.4673(2)	0.031(1)	0.034(1)	0.030(1)	–0.004(1)	0.010(1)	–0.001(1)
C(11)	8*f*	0.3862(1)	0.7508(3)	0.4320(2)	0.037(2)	0.051(2)	0.046(2)	–0.002(1)	0.018(1)	0.005(2)
C(12)	8*f*	0.3751(1)	0.8867(3)	0.4148(2)	0.051(2)	0.050(2)	0.056(2)	–0.013(2)	0.017(2)	0.014(2)
C(13)	8*f*	0.3381(1)	0.9503(3)	0.4328(2)	0.059(2)	0.036(2)	0.050(2)	–0.003(2)	0.010(2)	0.005(1)
C(14)	8*f*	0.3117(1)	0.8809(3)	0.4682(2)	0.061(2)	0.040(2)	0.052(2)	0.009(2)	0.023(2)	0.001(2)
C(15)	8*f*	0.3226(1)	0.7449(3)	0.4857(2)	0.049(2)	0.038(2)	0.043(2)	0.002(1)	0.022(2)	0.006(1)
C(16)	8*f*	0.3536(1)	0.4678(2)	0.5650(2)	0.037(2)	0.030(1)	0.030(2)	0.006(1)	0.012(1)	0.002(1)
C(17)	8*f*	0.3843(1)	0.5289(3)	0.6388(2)	0.049(2)	0.039(2)	0.039(2)	–0.002(1)	0.016(2)	–0.002(1)
C(18)	8*f*	0.3703(1)	0.5166(3)	0.7009(2)	0.069(2)	0.045(2)	0.033(2)	0.006(2)	0.019(2)	–0.005(1)
C(19)	8*f*	0.3265(1)	0.4410(3)	0.6919(2)	0.073(2)	0.055(2)	0.047(2)	0.012(2)	0.038(2)	0.007(2)
C(20)	8*f*	0.2971(1)	0.3769(3)	0.6209(2)	0.051(2)	0.053(2)	0.052(2)	0.001(2)	0.028(2)	0.009(2)
C(21)	8*f*	0.3101(1)	0.3905(3)	0.5575(2)	0.044(2)	0.042(2)	0.034(2)	–0.001(1)	0.015(1)	0.003(1)
C(22)	8*f*	0.3236(1)	0.4203(2)	0.3958(2)	0.038(2)	0.032(1)	0.028(1)	–0.004(1)	0.013(1)	0.004(1)
C(23)	8*f*	0.2708(1)	0.4617(3)	0.3594(2)	0.038(2)	0.044(2)	0.036(2)	0.002(1)	0.011(1)	0.002(1)
C(24)	8*f*	0.2351(1)	0.4018(3)	0.2894(2)	0.041(2)	0.052(2)	0.042(2)	–0.002(1)	0.006(1)	0.007(2)
C(25)	8*f*	0.2518(1)	0.3014(3)	0.2545(2)	0.059(2)	0.047(2)	0.031(2)	–0.014(2)	0.009(2)	0.002(1)
C(26)	8*f*	0.3039(1)	0.2591(3)	0.2893(2)	0.065(2)	0.040(2)	0.044(2)	–0.008(2)	0.026(2)	–0.009(1)
C(27)	8*f*	0.3397(1)	0.3185(3)	0.3596(2)	0.044(2)	0.036(2)	0.039(2)	–0.004(1)	0.016(1)	0.000(1)

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