Crystal structure of bis[(triphenylphospine-κP)(3-amino-5,6-dimethyl-1,2,4-triazine-κN^2)(μ-bromo)copper(I)], $[CuBr(C_5H_8N_4)(PC_{18}H_{15})]_2$

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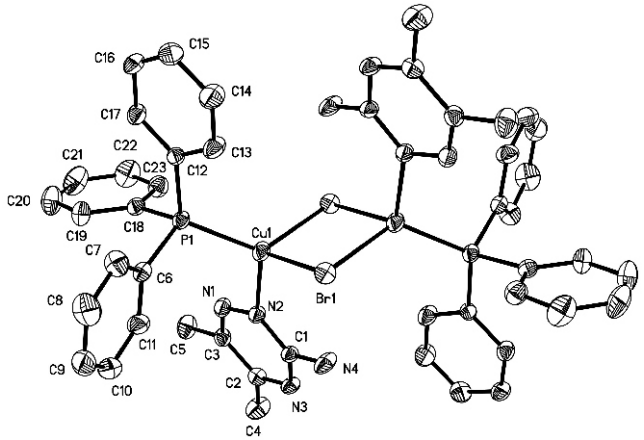

Abstract

$C_{46}H_{46}Br_2Cu_2N_8P_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.8813(8) Å, b = 10.0739(9) Å, c = 13.650(1) Å, α = 95.654(1)°, β = 107.957(2)°, γ = 113.160(1)°, V = 1150.3 Å^3, Z = 1, $R_{gt}(F)$ = 0.034, $wR_{ref}(F^2)$ = 0.083, T = 298 K.

Source of material

CuBr (0.0297 g, 0.3 mmol), triphenylphosphine (PPh_3, 0.0787 g, 0.3 mmol) and ADMT (ADMT = 3-amino-5,6-dimethyl-1,2,4-triazine, 0.0372 g, 0.3 mmol) were dissolved in dichloromethane (7 ml) and acetonitrile (3 ml), stirred for 3 h at room temperature, then filtered. Yellow crystals were obtained from the filtrate after standing at room temperature for several days.

Discussion

Copper(I) complexes have been studied well because of their interesting coordination chemistry and potential applications [1,2]. However,due to their low solubility in organic solvents and their instability, it is difficult to synthesize copper(I) complexes. We have obtained a series of Cu(I) complexes containing PPh_3 and nitrogen ligands [3-6].

The crystal structure of the title complex has a planar Cu_2Br_2 ring. In the plane, the Cu···Cu distance of 3.446 Å is longer than the sum of van der Waals radii of two copper atoms (2.8 Å), indicating that in the title complex there is no metal-metal bonding interaction. Each copper atom is coordinated by one P atom from PPh_3, two Br atoms and one N atom from ADMT. The angles around Cu are in the range of 95.73(9)-127.83(3) Å, which indicates a distorted tetrahedral environment of Cu. The bond distances are d(Cu—Br) = 2.560 Å, d(Cu—P) = 2.215(1) Å and d(Cu—N) = 2.075(3) Å, which are similar to those observed in $[CuBr(pycn)(PPh_3)]_2$ [7] and $[CuBr(py)(PPh_3)]_2$ [8]. The angles are ∠Br–Cu–Br = 95.41(2)° and ∠Cu–Br–Cu = 84.59(2)°, which are close to those found in the two complexes previously mentioned. Two adjacent $[Cu(\mu\text{-}Br)(ADMT)(PPh_3)]_2$ units are connected via intermolecular hydrogen bond to an $R_2^2(8)$ arrangement formed by two amino groups and two N atoms from two ADMT, resulting in a zigzag chain. The distance N(donor)···N(acceptor) and angle ∠N–H···N in hydrogen bond N–H···N are 2.997 Å and 173.00°, respectively.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.38 × 0.40 × 0.43 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	27.73 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6028, 3989
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2859
$N(param)_{refined}$:	274
Programs:	SHELXS-97, SHELXL-97, SHELXTL [9]

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

Atom	Site	x	y	z	U_{iso}
H(4A)	2*i*	0.4587	0.7376	0.5457	0.064
H(4B)	2*i*	0.4416	0.8660	0.5079	0.064
H(4C)	2*i*	0.9917	1.2591	0.5098	0.094
H(4D)	2*i*	1.0153	1.3315	0.6247	0.094
H(4E)	2*i*	0.8561	1.2883	0.5288	0.094
H(5A)	2*i*	1.1888	1.0546	0.6892	0.088
H(5B)	2*i*	1.1884	1.2092	0.7165	0.088
H(5C)	2*i*	1.1739	1.1447	0.6024	0.088
H(7)	2*i*	0.7067	0.4760	0.9359	0.067
H(8)	2*i*	0.6728	0.5930	1.0737	0.088
H(9)	2*i*	0.7239	0.8391	1.0967	0.084
H(10)	2*i*	0.8147	0.9705	0.9831	0.078
H(11)	2*i*	0.8433	0.8542	0.8427	0.062
H(13)	2*i*	0.5190	0.3029	0.6997	0.060
H(14)	2*i*	0.4487	0.0604	0.7145	0.071
H(15)	2*i*	0.6412	–0.0136	0.7880	0.064
H(16)	2*i*	0.9049	0.1542	0.8486	0.057
H(17)	2*i*	0.9781	0.3992	0.8388	0.050
H(19)	2*i*	1.0871	0.7096	0.9566	0.059
H(20)	2*i*	1.3570	0.8062	0.9966	0.081
H(21)	2*i*	1.4507	0.8059	0.8641	0.090
H(22)	2*i*	1.2773	0.7137	0.6878	0.087
H(23)	2*i*	1.0062	0.6238	0.6461	0.061

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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)	2*i*	0.66730(5)	0.60978(5)	0.61220(4)	0.0425(3)	0.0345(3)	0.0405(3)	0.0165(2)	0.0061(2)	0.0154(2)
Br(1)	2*i*	0.36644(4)	0.48421(4)	0.57679(3)	0.0367(2)	0.0364(2)	0.0459(3)	0.0112(2)	0.0190(2)	0.0140(2)
N(1)	2*i*	0.9059(3)	0.9090(3)	0.6408(2)	0.035(2)	0.039(2)	0.042(2)	0.015(2)	0.007(2)	0.011(2)
N(2)	2*i*	0.7464(3)	0.8327(3)	0.6096(2)	0.032(2)	0.027(2)	0.041(2)	0.011(1)	0.008(1)	0.010(1)
N(3)	2*i*	0.7159(3)	1.0362(3)	0.5470(2)	0.037(2)	0.030(2)	0.052(2)	0.016(2)	0.017(2)	0.019(2)
N(4)	2*i*	0.5009(3)	0.8255(3)	0.5363(3)	0.035(2)	0.032(2)	0.086(3)	0.014(2)	0.012(2)	0.029(2)
P(1)	2*i*	0.8026(1)	0.5695(1)	0.75912(8)	0.0340(5)	0.0305(5)	0.0346(6)	0.0134(4)	0.0090(4)	0.0140(4)
C(1)	2*i*	0.6568(4)	0.8983(4)	0.5656(3)	0.033(2)	0.027(2)	0.040(2)	0.014(2)	0.012(2)	0.010(2)
C(2)	2*i*	0.8704(4)	1.1085(4)	0.5777(3)	0.043(2)	0.034(2)	0.039(2)	0.015(2)	0.019(2)	0.012(2)
C(3)	2*i*	0.9694(4)	1.0435(4)	0.6266(3)	0.035(2)	0.036(2)	0.038(2)	0.012(2)	0.011(2)	0.009(2)
C(4)	2*i*	0.9396(5)	1.2605(4)	0.5585(4)	0.051(3)	0.045(2)	0.094(4)	0.017(2)	0.031(3)	0.036(3)
C(5)	2*i*	1.1458(4)	1.1198(4)	0.6618(3)	0.041(3)	0.061(3)	0.062(3)	0.013(2)	0.014(2)	0.021(2)
C(6)	2*i*	0.7784(4)	0.6516(4)	0.8741(3)	0.032(2)	0.040(2)	0.038(2)	0.017(2)	0.011(2)	0.012(2)
C(7)	2*i*	0.7278(5)	0.5757(5)	0.9443(3)	0.059(3)	0.054(3)	0.052(3)	0.020(2)	0.024(2)	0.015(2)
C(8)	2*i*	0.7077(6)	0.6460(6)	1.0272(4)	0.079(4)	0.092(4)	0.057(3)	0.032(3)	0.042(3)	0.025(3)
C(9)	2*i*	0.7385(6)	0.7924(6)	1.0412(4)	0.076(4)	0.100(4)	0.048(3)	0.056(3)	0.022(3)	0.007(3)
C(10)	2*i*	0.7912(6)	0.8700(5)	0.9729(4)	0.083(4)	0.066(3)	0.056(3)	0.050(3)	0.020(3)	0.013(3)
C(11)	2*i*	0.8096(5)	0.8008(4)	0.8894(3)	0.066(3)	0.051(3)	0.049(3)	0.036(2)	0.023(2)	0.019(2)
C(12)	2*i*	0.7557(4)	0.3779(3)	0.7699(3)	0.040(2)	0.027(2)	0.031(2)	0.014(2)	0.008(2)	0.010(2)
C(13)	2*i*	0.5977(5)	0.2746(4)	0.7312(3)	0.042(2)	0.038(2)	0.063(3)	0.017(2)	0.010(2)	0.022(2)
C(14)	2*i*	0.5556(5)	0.1286(4)	0.7392(4)	0.047(3)	0.039(2)	0.075(3)	0.011(2)	0.012(2)	0.020(2)
C(15)	2*i*	0.6701(5)	0.0844(4)	0.7830(3)	0.064(3)	0.037(2)	0.056(3)	0.019(2)	0.019(2)	0.019(2)
C(16)	2*i*	0.8270(5)	0.1846(4)	0.8195(3)	0.059(3)	0.048(2)	0.044(2)	0.033(2)	0.015(2)	0.022(2)
C(17)	2*i*	0.8710(4)	0.3316(4)	0.8135(3)	0.042(2)	0.042(2)	0.039(2)	0.018(2)	0.011(2)	0.015(2)
C(18)	2*i*	1.0181(4)	0.6560(4)	0.7973(3)	0.040(2)	0.033(2)	0.037(2)	0.018(2)	0.015(2)	0.015(2)
C(19)	2*i*	1.1247(5)	0.7110(4)	0.9019(3)	0.039(2)	0.060(3)	0.041(3)	0.018(2)	0.012(2)	0.015(2)
C(20)	2*i*	1.2863(5)	0.7680(5)	0.9260(4)	0.037(3)	0.084(3)	0.063(3)	0.015(2)	0.009(2)	0.019(3)
C(21)	2*i*	1.3418(5)	0.7685(6)	0.8473(5)	0.033(3)	0.097(4)	0.095(4)	0.026(3)	0.024(3)	0.039(3)
C(22)	2*i*	1.2382(6)	0.7137(5)	0.7417(4)	0.058(3)	0.102(4)	0.078(4)	0.040(3)	0.044(3)	0.037(3)
C(23)	2*i*	1.0761(5)	0.6588(4)	0.7169(3)	0.053(3)	0.058(3)	0.047(3)	0.027(2)	0.020(2)	0.018(2)

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