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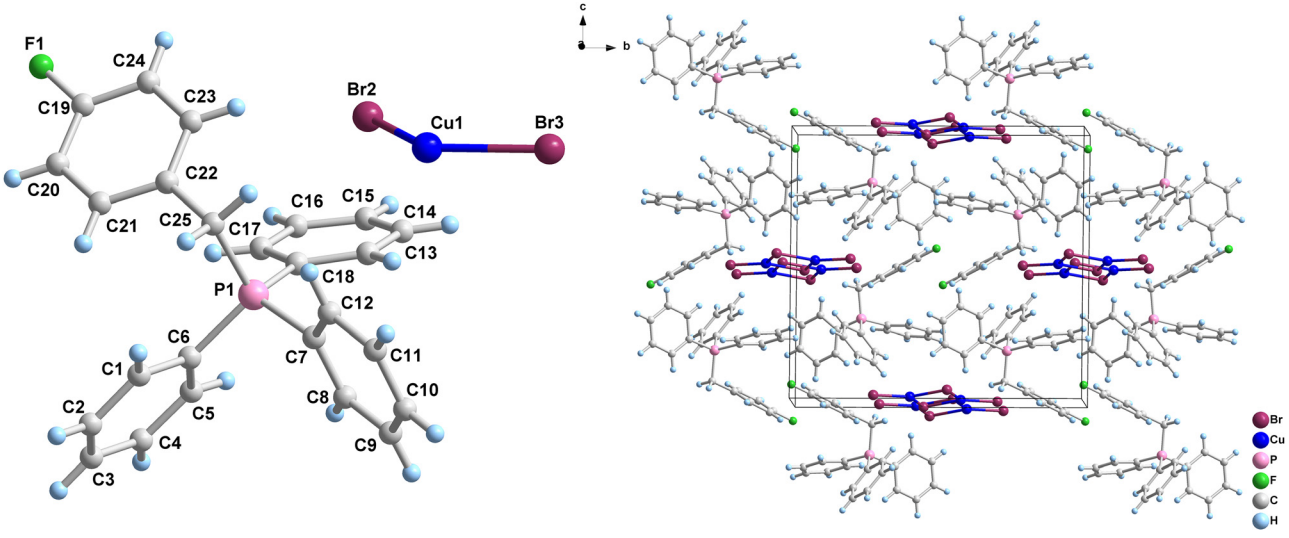
Crystal structure of {[(4-fluorophenyl)methyl] triphenylphosphonium}dibromocopper(I), $[C_{25}H_{21}FP]^{+}[CuBr_2]^{-}$



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Abstract

$C_{25}H_{21}Br_2CuFP$, monoclinic, $P2_1/c$ (no. 14), a = 8.8442(2) Å, b = 16.8942(3) Å, c = 16.0626(3) Å, β = 98.332(2)°, V = 2374.67(8) Å^3, Z = 4, $R_{gt}(F)$ = 0.0565, $wR_{ref}(F^2)$ = 0.1609, T = 293(2).

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.10 × 0.08 × 0.06 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	6.02 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, Dualflex, HyPix, ω
θ_{max}, completeness:	74.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25358, 4734, 0.069
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3,791
$N(param)_{refined}$:	271
Programs:	CrysAlis^{PRO},[1] SHELX,[2,4] Olex2,[3] Diamond[5]

1 Source of materials

The [(4-fluorophenyl)methyl]triphenylphosphonium bromide (**4FBTP**) (0.112 g, 0.25 mmol) and CuBr (0.035 g, 0.25 mmol) were added in 0.5 mL HBr and 3.5 mL CH_2Cl_2/CH_3CH_2OH (*v*/*v*, 4/3) to form a clear colorless solution. Then, 250 µL hypophosphorous acid was added in reaction system to prevent Cu^+ from oxidation. After stirred for 20 min, the mixed solution was filtered using nylon-66 membrane syringe filter and maintained at room temperature to obtain some block crystals after 15 days.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br2	0.82171 (8)	0.79130 (4)	0.49411 (5)	0.0883 (3)
Br3	1.19863 (8)	0.95622 (4)	0.52493 (4)	0.0807 (2)
C1	0.2358 (4)	0.8172 (2)	0.7252 (3)	0.0436 (8)
H1	0.238676	0.851394	0.680097	0.052*
C2	0.1132 (5)	0.8187 (3)	0.7696 (3)	0.0541 (10)
H2	0.033269	0.853884	0.754072	0.065*
C3	0.1083 (5)	0.7685 (3)	0.8366 (3)	0.0549 (11)
H3	0.025372	0.769872	0.866195	0.066*
C4	0.2261 (6)	0.7163 (3)	0.8598 (3)	0.0551 (11)
H4	0.221619	0.681856	0.904520	0.066*
C5	0.3519 (5)	0.7145 (3)	0.8169 (2)	0.0466 (9)
H5	0.432792	0.680194	0.833885	0.056*
C6	0.3556 (4)	0.7640 (2)	0.7487 (2)	0.0358 (7)
C7	0.6291 (4)	0.8514 (2)	0.7277 (2)	0.0373 (7)
C8	0.6771 (5)	0.8575 (3)	0.8135 (3)	0.0473 (9)
H8	0.652581	0.818061	0.849674	0.057*
C9	0.7615 (5)	0.9226 (3)	0.8453 (3)	0.0572 (11)
H9	0.794286	0.926839	0.902782	0.069*
C10	0.7967 (5)	0.9810 (3)	0.7916 (3)	0.0595 (11)
H10	0.852081	1.025071	0.813064	0.071*
C11	0.7510 (6)	0.9746 (3)	0.7068 (3)	0.0600 (12)
H11	0.776532	1.014167	0.670953	0.072*
C12	0.6668 (5)	0.9097 (2)	0.6737 (3)	0.0488 (9)
H12	0.636070	0.905342	0.616059	0.059*
C13	0.7845 (5)	0.6837 (3)	0.7230 (4)	0.0577 (11)
H13	0.832066	0.732339	0.718656	0.069*
C14	0.8715 (6)	0.6158 (3)	0.7411 (5)	0.0799 (18)
H14	0.977613	0.619077	0.749534	0.096*
C15	0.8010 (6)	0.5436 (3)	0.7466 (4)	0.0688 (14)
H15	0.859732	0.498563	0.760328	0.083*
C16	0.6463 (6)	0.5379 (3)	0.7320 (3)	0.0602 (11)
H16	0.599958	0.488686	0.734133	0.072*
C17	0.5573 (5)	0.6046 (2)	0.7142 (3)	0.0488 (9)
H17	0.451368	0.600259	0.704001	0.059*
C18	0.6268 (4)	0.6787 (2)	0.7114 (2)	0.0374 (7)
C19	0.2154 (7)	0.5700 (3)	0.4782 (3)	0.0634 (13)
C20	0.1375 (6)	0.6233 (3)	0.5185 (3)	0.0601 (12)
H20	0.035356	0.615022	0.523724	0.072*
C21	0.2134 (5)	0.6906 (3)	0.5519 (3)	0.0486 (9)
H21	0.161302	0.728212	0.578992	0.058*
C22	0.3661 (4)	0.7020 (2)	0.5451 (2)	0.0399 (8)
C23	0.4420 (5)	0.6463 (3)	0.5039 (3)	0.0511 (9)
H23	0.544743	0.653414	0.499337	0.061*
C24	0.3656 (7)	0.5794 (3)	0.4693 (3)	0.0634 (13)
H24	0.415534	0.541930	0.440658	0.076*
C25	0.4490 (4)	0.7751 (2)	0.5802 (2)	0.0390 (7)
H25A	0.381121	0.820303	0.570474	0.047*
H25B	0.535715	0.784505	0.550829	0.047*
Cu1	0.93376 (15)	0.91444 (6)	0.49641 (6)	0.0938 (4)
F1	0.1406 (5)	0.5042 (2)	0.4450 (2)	0.0967 (12)
P1	0.51548 (9)	0.76650 (5)	0.69154 (5)	0.0328 (2)

2 Experimental details

The data were collected using Rigaku,[1] the structure of crystal were solved by SHELXT program within Olex2 and refined by SHELXL.[2–4] The graphics were finished using Diamond.[5]

3 Comment

In this investigation a copper(I)-based metal halide was prepared by the slow evaporation. As is shown in the left part of the figure, the asymmetric unit of title compound contains a [(4-fluorophenyl)methyl]triphenylphosphonium (**4FBTP**) cation, $[CuBr_2]^-$ anionic moiety, which forms a $[Cu_2\text{-}\mu^{2-}Br_2Br_2]^{2-}$ anion located around an inversion center. The bond distances of aromatic rings of **4FBTP** are in normal range.[6] And the distances of Cu1–Br2, Cu1–Br3 are 2.302 Å, 2.426 Å within the normal range of the literatures which have been reported.[7–9] Above all, the packing of the structure consists of **4FBTP** cations and $[Cu_2Br_4]^{2-}$ anions. (*cf.* right part of the figure).

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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