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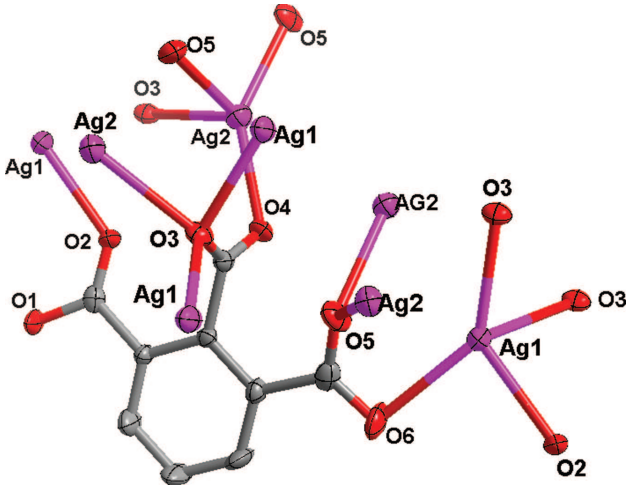
Crystal structure of poly[μ_8-3-carboxyphthalat-κ^8-*O*:*O*^{1},*O*^{1},*O*^{1}:*O*^{2}:*O*^{3},*O*^{3}:*O*^{4})silver(I)], $C_9H_4Ag_2O_6$



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Abstract

$C_9H_4Ag_2O_6$, monoclinic, $P2_1/c$ (no. 14), $a = 13.8224(6)$ Å, $b = 9.5222(4)$ Å, $c = 7.2104(3)$ Å, $\beta = 101.991(4)°$, $V = 928.32(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0239$, $wR_{ref}(F^2) = 0.0411$, $T = 293(2)$ 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.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.18 × 0.17 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	4.23 mm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, Gemini, ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4331, 1626, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1311
$N(param)_{refined}$:	155
Programs:	CrysAlis^{PRO} [1], SHELX [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
AG1	0.40346(3)	−0.47118(4)	0.14994(5)	0.02744(11)
AG2	0.50829(3)	0.18578(4)	−0.03486(5)	0.03102(12)
O1	0.1804(2)	0.3955(3)	0.2451(5)	0.0369(9)
H1	0.204684	0.472342	0.231393	0.055*
O2	0.3174(2)	0.3165(3)	0.1614(4)	0.0269(8)
O3	0.4317(2)	0.0836(3)	0.3533(4)	0.0252(7)
O4	0.3885(2)	0.0425(3)	0.0425(4)	0.0262(7)
O5	0.3817(2)	−0.2122(3)	0.2662(5)	0.0360(9)
O6	0.2483(2)	−0.3451(3)	0.1882(5)	0.0430(9)
C1	0.2005(3)	0.1496(4)	0.2228(6)	0.0183(10)
C2	0.2636(3)	0.0344(4)	0.2210(6)	0.0167(10)
C3	0.2248(3)	−0.1013(4)	0.2265(6)	0.0215(10)
C4	0.1270(3)	−0.1174(5)	0.2384(7)	0.0360(13)
H4	0.101438	−0.207480	0.241973	0.043*
C5	0.0665(4)	−0.0040(5)	0.2452(8)	0.0446(16)
H5	0.001017	−0.017239	0.254601	0.054*
C6	0.1038(3)	0.1292(5)	0.2381(7)	0.0332(13)
H6	0.063249	0.206441	0.243536	0.040*
C7	0.2399(3)	0.2950(4)	0.2086(6)	0.0212(10)
C8	0.3698(3)	0.0560(4)	0.2047(7)	0.0205(10)
C9	0.2895(4)	−0.2289(5)	0.2285(6)	0.0274(12)

Source of materials

An excess of aqueous NH_3 solution was slowly added dropwise to a suspension of Ag_2O (0.023 g, 0.1 mmol) in MeOH/H_2O (6 mL, 5:1 v/v), and the mixture was stirred for 15 min. 1,2,3-H3btc (benzene-1,2,3-tricarboxylic acid, 0.021 g, 0.1 mmol) was then slowly added, and stirring was continued for another 30 min. The resultant colorless solution was

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allowed to stand in the dark at room temperature for a week to give colorless block crystals.

Experimental details

Absorption corrections were applied by using multi-scan program. Hydrogen atoms attached to C of the title complex located in difference electron density maps, and treated as riding atoms. The U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C).

Comment

The metallophilicity is widespread in all coinage metal (Cu(I), Ag(I), and Au(I)) complexes [3–5]. Among them, the argentophilic interaction, the aggregation between Ag centers, has been used to facilitate charming silver-organic architectures. Recently, a buckyball-like Ag180 nanocage, constructed by 360 Ag···Ag edges was reported [6]. The argentophilic interactions play important roles in fabricating not only silver clusters or cages but also new silver-organic frameworks [7, 8]. Aiming to generate new silver-organic frameworks based on diverse argentophilic interactions, we choose 1,2,3-benzenetricarboxylic acid (H_3btc) as educt. This benzenepolycarboxylic acid ligands possess ortho carboxylic groups, which were facilitated to form a rich variety of coordination modes for completely or partially deprotonated and abundant Ag···Ag interactions.

Single-crystal X-ray diffraction analysis reveals that the title complex exhibits a two-dimensional coordination network. In each asymmetric unit, there are two Ag(I) ion and one partially deprotonated $Hbtc^{2-}$ anion. Taking weak Ag···O interactions into account (up to 2.7 Å), each $Hbtc^{2-}$ anion coordinates to eight Ag(I) ions with its three functional groups. The carboxylate group in the 2-position shows tetradentate bridging modes, while those in the 3-positions exhibit tridentate bridging modes. It is noteworthy that Ag···Ag interactions fall into the range of 3.159(5)–3.174(6) Å, which are not exceptional and agree with the previously reported values. The Ag(I) ions aggregate into a layer parallel to the *bc* plane with the phenyl groups protrude from both sides, which is comprised of tetranuclear and octanuclear rings. All these extensive Ag···O interactions reinforce the complicated 2D layer. Adjacent 2D layers are bridged through very weak hydrogen bonds (C—H···O, H5···O1 2.642(2) Å) to give rise to three-dimensional supramolecular network.

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