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Crystal structure of $(\mu_4$ -(1,2,4,5-tetra(1,2,4-triazol-1-ylmethyl)-benzene- $\kappa^4 N:N^1:N^2:N^3$)disilver(I) diperchlorate

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.25 × 0.23 × 0.22 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.74 mm ⁻¹
Diffractometer, scan mode:	Rigaku Saturn 724, ω
$\theta_{\max}$, completeness:	27.9°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10,828, 3175, 0.031
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2566
$N(\text{param})_{\text{refined}}$:	184
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

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Abstract

$\text{C}_9\text{H}_9\text{N}_6\text{O}_4\text{ClAg}$, monoclinic, $C2/c$ (no. 15), $a = 24.730(5)$ Å, $b = 11.776(2)$ Å, $c = 9.6860(19)$ Å, $\beta = 109.09(3)^\circ$, $V = 2665.6(9)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0646$, $wR_{\text{ref}}(F^2) = 0.2177$, $T = 293(2)$ K.

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

Source of material

1,2,4,5-Tetrakis(1,2,4-triazol-1-ylmethyl)-benzene (0.04 g, 0.1 mmol) and AgNO_3 (0.034 mg, 0.20 mmol) were added to the mixture of water (7 mL) and acetonitrile (2 mL) which also contained perchloric acid in a Teflon-lined stainless steel reactor. The mixture was heated at 383 K for three

days, and then slowly cooled down to room temperature. Colorless crystals of the title compound were obtained.

Experimental details

The crystallographic data of title complex was collected on a Rigaku Saturn 724 CCD diffractometer at room temperature. Absorption corrections were applied by using multi-scan program [1]. Using Olex2 [2], the structure was solved with the ShelXT [3] structure solution program using Intrinsic Phasing and refined with the ShelXL [4] refinement package. The H atoms were fixed, with C–H distance of 0.93 Å; and/or positioned geometrically in the riding-model approximation, with C–H distance of 0.97 Å; $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Comment

The architectural design and construction of coordination polymers (CPs) have received much attention since the wide variety of potential applications are closely related to the diverse structures [5–8]. Among them, CPs based on silver(I) have attracted extensive attention because of their potential applications for anion exchange and light-emitting diodes [9–11]. So far, nitrogen-containing ligands such as 1,10-phenanthroline, pyrazine, bipyridine, bis(imidazole), tris(imidazol), and tetra(imidazol) have been employed in

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} ^a /U _{eq}
Ag1	0.21677 (2)	0.37284 (5)	0.42998 (6)	0.0612 (3)
N1	0.1355 (2)	0.3539 (5)	0.2647 (6)	0.0496 (12)
N2	0.0706 (2)	0.3596 (4)	0.0516 (5)	0.0446 (11)
N3	0.0410 (2)	0.3522 (5)	0.1473 (6)	0.0518 (12)
N4	0.10433 (19)	−0.0715 (4)	−0.1115 (4)	0.0378 (9)
N5	0.1147 (2)	−0.0677 (5)	−0.2408 (5)	0.0479 (11)
N6	0.1953 (2)	−0.1009 (5)	−0.0545 (6)	0.0492 (12)
C1	0.0818 (3)	0.3488 (5)	0.2747 (6)	0.0462 (12)
H1	0.074685	0.343521	0.363096	0.055*
C2	0.1258 (3)	0.3610 (5)	0.1238 (7)	0.0515 (14)
H2	0.154175	0.366411	0.080488	0.062*
C3	0.0408 (3)	0.3694 (5)	−0.1048 (7)	0.0495 (14)
H3A	0.066509	0.402962	−0.150622	0.059*
H3B	0.008509	0.420202	−0.120647	0.059*
C4	0.0196 (2)	0.2570 (4)	−0.1773 (5)	0.0362 (10)
C5	0.0390 (2)	0.1545 (4)	−0.1133 (6)	0.0353 (10)
H5	0.065875	0.154432	−0.020022	0.042*
C6	0.02064 (19)	0.0509 (4)	−0.1803 (5)	0.0352 (10)
C7	0.0474 (2)	−0.0556 (5)	−0.1020 (6)	0.0427 (12)
H7A	0.049752	−0.050815	−0.000173	0.051*
H7B	0.023648	−0.120408	−0.145107	0.051*
C8	0.1524 (2)	−0.0925 (5)	−0.0037 (6)	0.0460 (13)
H8	0.155381	−0.100284	0.094009	0.055*
C9	0.1697 (3)	−0.0857 (7)	−0.2021 (7)	0.0544 (15)
H9	0.189743	−0.088025	−0.268342	0.065*
Cl1	0.32307 (14)	0.1760 (3)	0.2413 (3)	0.1094 (9)
O1 ^a	0.3484 (6)	0.2746 (11)	0.1906 (14)	0.1281 (16)
O2 ^a	0.3657 (6)	0.1304 (11)	0.3689 (13)	0.1281 (16)
O3 ^a	0.3051 (6)	0.0947 (11)	0.1251 (14)	0.1281 (16)
O4 ^a	0.2745 (5)	0.2182 (12)	0.2788 (14)	0.1281 (16)
O1A ^a	0.3004 (6)	0.0942 (11)	0.3172 (14)	0.1281 (16)
O2A ^a	0.2857 (6)	0.2682 (11)	0.2097 (15)	0.1281 (16)
O3A ^a	0.3769 (5)	0.2074 (12)	0.3351 (14)	0.1281 (16)
O4A ^a	0.3269 (6)	0.1246 (11)	0.1162 (14)	0.1281 (16)

^aOccupancy: 0.5.

the construction of silver(I) CPs [12–15]. Here, we choose the flexible tetrapodal 1,2,4,5-tetra(1,2,4-triazol-1-ylmethyl)-benzene (ttyb) as a functional ligand. Comparing the title ligand with the imidazole groups in the tetrapodal ligand 1,2,4,5-tetrakis(imidazol-1-ylmethyl)benzene, the triazole groups in ttyb could provide the possibility of transferring the coordination modes from 1 to 2 aggregates during the reaction, which may initiate the structural transformation.

X-ray analysis reveals that title complex crystallizes in monoclinic space group *C2/c*. The asymmetric unit of title complex contains one independent Ag(I) cation, one half of ttyb ligand (located on a twofold axis), and one perchlorate anion (see the figure; the asymmetric unit is labeled and perchlorate anions are omitted for clarity). Each Ag(I)

center is coordinated by two nitrogen atoms from two L ligands, showing an approximately linear coordination environment. The N–Ag–N bond angles are all 167.18°. The Ag–N lengths are in the range of 2.130(18)–2.145(2) Å. In the structure of the title compound the perchlorate anions do not show any bonding interactions with Ag(I) cations, only acting as counteranions. The Ag(I) cations are connected by the ttyb ligands to form a 2D layer wave-like structure with the porous diameter of 23.4 × 6.5 Å. From a topological perspective, the L ligands can be viewed as four-connected nodes, and the Ag(I) cations can be considered as linkers, thus this 2D layer is topologically equivalent to a four-connected square layer.

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