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Crystal structure of catena-(μ_2-4,4'-bipyridine)silver(I)hydrogen-*bis*-(5-nitro-1,2-carboxybenzene-3-carboxylato)tetrahydrate, $[Ag(C_{10}H_8N_2)]\cdot(C_{18}H_9O_{16}N_2)\cdot 4H_2O$, $C_{28}H_{25}AgN_4O_{20}$

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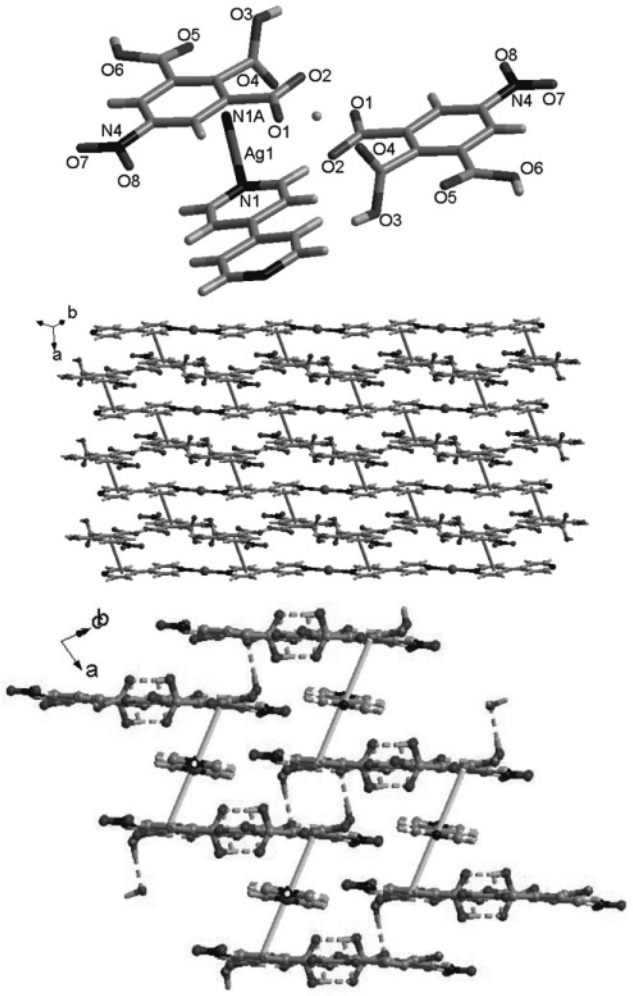

Abstract

$C_{28}H_{25}AgN_4O_{20}$, triclinic, $P\bar{1}$ (no. 2), a = 8.3973(9) Å, b = 9.519(1) Å, c = 11.273(1) Å, α = 66.089(1)°, β = 82.176(1)°, γ = 78.375(1)°, V = 805.3 Å^3, Z = 1, $R_{gt}(F)$ = 0.0244, $wR_{ref}(F^2)$ = 0.0686, T = 296 K.

Source of material

A mixture of $AgNO_3$ (17.0 mg, 0.1 mmol), bipy (17.9 mg, 0.1 mmol), 5-nitro-1,2,3-benzenecarboxylic acid (H_3nbtc) (25.2 mg, 0.1 mmol), KOH (11.2 mg, 0.2 mmol) in H_2O (12 mL) was sealed into a 25 mL stainless steel reactor with a teflon liner and heated at 443 K for 3 days. The reactor was then cooled slowly to room temperature and the solution filtered. Colourless block-shaped crystals were obtained from the filtrate after several days at room temperature.

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Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.11×0.18×0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.23 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6163, 2983
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2898
$N(param)_{refined}$:	242
Programs:	SHELX [16]

Discussion

The design and construction of metal-organic coordination polymers has been shown to be a promising field, not only because of their potential applications as functional solid materials in ion exchange, catalysis, luminescence, magnets, nonlinear optics, and gas separation/storage [1-6]. Among various organic ligands, rodlike *N*,*N*′-donor building blocks, such as the conventionally employed 4,4'-bipyridine (bipy), are widely employed to construct such coordination polymers [3,7-11]. $[Ag(\mu_2\text{-bipy})]^+$ complexes have been extensively studied in coordination chemistry [12-15]. Major reasons for this interest emergefrom its excellent photo-luminescent property. It may be used to assemble supramolecular architectures via π-π-aromatic interactions with other organic bridge-ligands. Herein, we reported a new three-dimensional supramolecular coordination polymeric network based on $[Ag(\mu_2\text{-bipy})]^+$ with 5-nitro-1,2,3-benzenecarboxylic acid (H_3nbtc). In the title complex, there are two Ag(I) ions, one 4,4'-bipyridine, one hydrogen-*bis*-(5-nitro-1,2-carboxybenzene-3-carboxylato), and four free water molecules. Ag1 is coordiated by two pyridine nitrogen atoms from different 4,4'-bipyridine molecules showing linear coordination geometry with an N–Ag–N bond angle of 180.0 °. The bond lengths of Ag–N are all 2.1646(16) Å. O4 shows weak coordination to Ag1 with an Ag···O distance of 2.826 Å. Clearly, π-π-aromatic interactions and hydrogen bonds play a vital role in the formation of the structure. Aromatic $\pi\cdots\pi$ stacking interactions exist between the benzene ring and the pyridine ring to form a two-dimensional layers. The angle between the benzene ring plane and pyridine ring plane are 7.75(6) °. The distance of Cg1···Cg2 are 3.84 Å; Cg1 and Cg2 are the centroids of benzene ring C1-C6, and pyridine ring C10-C14 and N1, respectively (Figure middle). There are three kinds of hydrogen bonds: (a) hydrogen bonding between the free water molecules and the oxygen of the hydrogen-*bis*-(5-nitro-1,2-carboxy- benzene-3-carboxylato) ions with d(O···O) = 3.033(3) Å, 2.557(2) Å, 2.907(3) Å, 2.883(3) Å; (b) hydrogen bonding between the free water molecules with d(O···O) = 2.738(3) Å,

3.150(3) Å; (c) hydrogen bonding between the hydrogen-*bis*-(5-nitro-1,2-carboxybenzene-3-carboxylato) ions with d(O···O) = 2.598(2) Å, 2.453(3) Å, respectively (Figure bottom). A three-dimensional supramolecular architecture of π-π-aromatic interactions and hydrogen bonds contributes to the stability of the structure.

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

Atom	Site	x	y	z	U_{iso}
H(1D)	1*e*	½	½	0	0.082
H(3)	2*i*	0.4033	0.1005	0.5222	0.066
H(6)	2*i*	0.7331	0.2451	0.8101	0.067
H(1W)	2*i*	0.3854	0.0633	0.1572	0.114
H(2W)	2*i*	0.4102	0.2027	0.1194	0.114
H(3W)	2*i*	0.3822	0.8713	–0.0065	0.100
H(4W)	2*i*	0.2648	0.9752	0.0190	0.100
H(4)	2*i*	0.8179	0.5236	0.5404	0.034
H(6A)	2*i*	0.6998	0.6564	0.1721	0.037
H(10)	2*i*	0.1532	0.3003	0.3458	0.046
H(11)	2*i*	0.1450	0.5080	0.1508	0.044
H(13)	2*i*	–0.1211	0.2988	0.0243	0.048
H(14)	2*i*	–0.1088	0.0973	0.2234	0.050

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}
Ag(1)	1*b*	0	0	½	0.0474(2)	0.0316(1)	0.0222(1)	–0.01076(9)	–0.00576(9)	0.00628(9)
O(1)	2*i*	0.5619(2)	0.5282(2)	0.0787(2)	0.093(1)	0.0434(9)	0.0310(8)	–0.0303(9)	–0.0247(8)	–0.0024(7)
O(2)	2*i*	0.4857(2)	0.3101(2)	0.2259(2)	0.076(1)	0.051(1)	0.0394(9)	–0.0360(9)	–0.0170(8)	–0.0095(8)
O(3)	2*i*	0.4277(2)	0.1860(2)	0.5054(2)	0.0409(8)	0.0347(8)	0.061(1)	–0.0144(6)	0.0081(7)	–0.0221(7)
O(4)	2*i*	0.6685(2)	0.0768(2)	0.4427(2)	0.0366(7)	0.0310(7)	0.057(1)	–0.0091(6)	0.0005(7)	–0.0198(7)
O(5)	2*i*	0.6731(3)	0.1263(2)	0.6863(2)	0.134(2)	0.0411(9)	0.0323(9)	–0.047(1)	–0.018(1)	0.0018(7)
O(6)	2*i*	0.7421(2)	0.3093(2)	0.7354(1)	0.070(1)	0.0418(8)	0.0243(7)	–0.0207(8)	–0.0068(7)	–0.0093(6)
O(7)	2*i*	0.9115(2)	0.7654(2)	0.3949(2)	0.0516(9)	0.0452(9)	0.0486(9)	–0.0237(7)	–0.0037(7)	–0.0219(7)
O(8)	2*i*	0.8527(4)	0.8329(2)	0.1986(2)	0.154(2)	0.059(1)	0.052(1)	–0.070(1)	–0.042(1)	0.018(1)
O(9)	2*i*	0.4052(3)	0.1379(2)	0.0862(2)	0.123(2)	0.051(1)	0.058(1)	–0.038(1)	0.008(1)	–0.017(1)
O(10)	2*i*	0.2860(3)	0.8804(2)	0.0277(2)	0.095(2)	0.059(1)	0.0373(9)	–0.022(1)	–0.0088(9)	–0.0045(8)
N(1)	2*i*	0.0197(2)	0.1791(2)	0.3067(2)	0.0398(9)	0.0292(8)	0.0216(8)	–0.0090(7)	–0.0045(6)	0.0007(6)
N(4)	2*i*	0.8493(2)	0.7459(2)	0.3120(2)	0.052(1)	0.0287(9)	0.040(1)	–0.0174(8)	–0.0094(8)	–0.0071(8)
C(1)	2*i*	0.6329(2)	0.4496(2)	0.2938(2)	0.0332(9)	0.0257(9)	0.0269(9)	–0.0058(7)	–0.0067(7)	–0.0101(7)
C(2)	2*i*	0.6381(2)	0.3396(2)	0.4221(2)	0.0292(9)	0.0235(9)	0.0278(9)	–0.0062(7)	–0.0034(7)	–0.0096(7)
C(3)	2*i*	0.7062(2)	0.3684(2)	0.5159(2)	0.0325(9)	0.0237(8)	0.0256(9)	–0.0060(7)	–0.0038(7)	–0.0082(7)
C(4)	2*i*	0.7731(2)	0.5032(2)	0.4792(2)	0.0349(9)	0.0270(9)	0.0281(9)	–0.0074(7)	–0.0066(7)	–0.0125(8)
C(5)	2*i*	0.7725(2)	0.6060(2)	0.3513(2)	0.0341(9)	0.0224(9)	0.033(1)	–0.0094(7)	–0.0046(8)	–0.0096(8)
C(6)	2*i*	0.7014(2)	0.5835(2)	0.2574(2)	0.040(1)	0.0258(9)	0.0247(9)	–0.0081(8)	–0.0062(8)	–0.0052(7)
C(7)	2*i*	0.5524(2)	0.4242(2)	0.1930(2)	0.040(1)	0.032(1)	0.032(1)	–0.0071(8)	–0.0104(8)	–0.0118(8)
C(8)	2*i*	0.5759(2)	0.1880(2)	0.4578(2)	0.034(1)	0.030(1)	0.031(1)	–0.0091(8)	–0.0039(8)	–0.0106(8)
C(9)	2*i*	0.7053(3)	0.2549(2)	0.6548(2)	0.042(1)	0.031(1)	0.027(1)	–0.0085(8)	–0.0051(8)	–0.0086(8)
C(10)	2*i*	0.0951(3)	0.3008(3)	0.2810(2)	0.046(1)	0.041(1)	0.024(1)	–0.0157(9)	–0.0108(8)	–0.0014(8)
C(11)	2*i*	0.0910(3)	0.4264(2)	0.1634(2)	0.047(1)	0.034(1)	0.027(1)	–0.0188(9)	–0.0095(8)	–0.0005(8)
C(12)	2*i*	0.0061(2)	0.4316(2)	0.0635(2)	0.0330(9)	0.0259(9)	0.0190(9)	–0.0073(7)	–0.0017(7)	–0.0029(8)
C(13)	2*i*	–0.0664(3)	0.3032(2)	0.0888(2)	0.062(1)	0.034(1)	0.024(1)	–0.021(1)	–0.0119(9)	–0.0026(8)
C(14)	2*i*	–0.0581(3)	0.1815(2)	0.2092(2)	0.065(1)	0.029(1)	0.029(1)	–0.021(1)	–0.010(1)	0.0001(8)

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