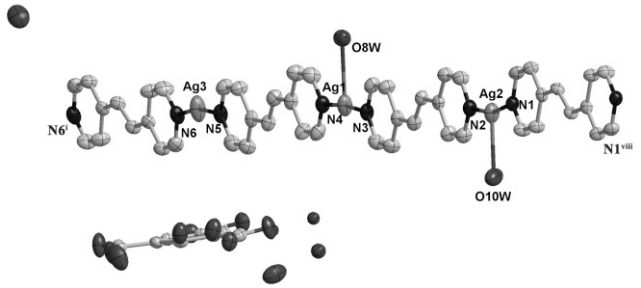
Crystal structure of di-aqua-tris(μ-1,2-bis-(4-pyridyl)ethylene-κN:κN')-trisilver(I)-benzene-1,3,5-tricarboxylate octahydrate, $[Ag_3(H_2O)_2(C_{12}H_{10}N_2)_3][C_9H_3O_6]\cdot 8H_2O$, $C_{90}H_{102}Ag_6N_{12}O_{30}$

Fu-An Li[*, I], Jin-Min Liu[II] and Wei-Chun Yang[I]

[I] College of Chemistry and Chemical Engineering, Pingdingshan University, Pingdingshan 467000, Henan Province, P. R. China
[II] Pingdingshan Finance and Economics School, Pingdingshan 467000, Henan Province, P. R. China

Received November 23, 2011, accepted April 17, 2012, available online June 28, 2012, CCDC no. 1267/3758



Abstract

$C_{90}H_{102}Ag_6N_{12}O_{30}$, triclinic, $P\bar{1}$ (no. 2), a = 10.5731(6) Å, b = 15.342(1) Å, c = 16.867(1) Å, α = 108.053(6)°, β = 93.184(5)°, γ = 108.118(5)°, V = 2437.2 Å^3, Z = 1, $R_{gt}(F)$ = 0.0454, $wR_{ref}(F^2)$ = 0.1467, T = 296 K.

Source of material

All chemicals and solvents used in the synthesis were of reagent grade and were used without further purification. Excess 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. Benzene-1,3,5-tricarboxylic acid (H_3btc, 0.021 g, 0.1 mmol) and 4,4'-bipyridine (0.015 g, 0.1 mmol) were then slowly added, and stirring was continued for another 30 min. The resultant colourless solution was allowed to stand in the dark at room temperature for a week to give colourless needles.

Table 1. Data collection and handling.

Crystal:	colourless needles, size 0.19×0.20×0.21mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.67 cm^{-1}
Diffractometer, scan mode:	Bruker Smart APEX II CCD, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18283, 8557
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6146
$N(param)_{refined}$:	631
Programs:	SHELXS-97, SHELXL-97 [9], DIAMOND [10]

Experimental details

Hydrogen atoms connected to carbon atoms were generated geometrically and refined as riding atoms with d(C–H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C).The approximate positions of the water H atoms, obtained from a difference Fourier map, were restrained to ideal configuration of the water molecule and fixed in the final stages of refinement with d(O–H) = 0.85 Å.

Discussion

The research on transition metal-organic coordination polymers has been rapidly expanding because of their intriguing topologies and potential applications, e.g. in catalysis, non-linear optics, fluorescence, electrical conductivity and magnetism, etc. [1-5]. As a result, the preparation of coordination polymers with fascinating structure has attracted considerable attention, and some coordination ploymers have been obtained and reported in recent years [6-7]. In the title compound, $\{[Ag_3(H_2O)_2(C_{12}H_{10}N_2)_3][C_9H_3O_6]\cdot 8H_2O]_2\}_n$, the asymmetric unit consists of three Ag^+ ions, two coordination water molecules, three μ-1,2-di(4-pyridyl)-ethylene-κN:κN'molecules, one benzene-1,3,5-tricarboxylate anion (bta^{3-}) and eight uncoordi nated water molecules. The Ag^+ ions are linked by bridging μ-1,2-di(4-pyridyl)-ethylene-κN:κN'molecules to form 1D chain structure. Ag3 is twofold-coordinated by two N atoms from two μ-1,2-di(4-pyridyl)-ethylene-κN:κN'molecules, with distances Ag3–N5 = 2.1374(49) Å and Ag3–N6 = 2.1429(28) Å and an angle of N6–Ag3–N5 = 167.646(171)°. The other two Ag^+ ions (Ag1, Ag2) are threefold-coordinated by two N atoms from two μ-1,2-di(4-pyridyl)-ethylene-κN:κN' molecules and one O atom from a water molecule with Ag2–N1 = 2.1467(46) Å, Ag2–N2 = 2.1412(49) Å, Ag2–O10w = 2.5612(35) Å, N2–Ag2–N1 = 164.739(165)°, Ag1–N3 = 2.1265(49) Å, Ag1–N4 = 2.1268(48) Å, Ag1–O8w = 2.7006(31) Å, N4–Ag1–N2 = 173.638(169)°, as commonly observed [8]. There are three kinds of hydrogen bonds in the complex structure. Firstly, there are four hydrogen bonds between coordinated water molecules ($O10_w$) and the bta^{3-} anion (O4, O2), $O10_w$–$H10_wA\cdots$O4, $O10_w$–$H10_wB\cdots$O2 (d(O···O) = 2.78Å, 2.78Å); Secondly, six hydrogen bonds have been formed by non-coordinated water molecules, and the O···O distances are in the range of 2.7Å-2.941Å; Thirdly, nine hydrogen bonds are observed between non-coordinated water molecules and the bta^{3-} anion, their distances are in the range of 2.747 - 2.847 Å. Through these hydrogen bonds, the chain molecules are connected with each other to form a 2D super-molecular structured coordination polymer.

* Correspondence author (e-mail: lifuanpds@163.com)

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(5)	2*i*		0.3409	0.3285	0.1877	0.036
H(7)	2*i*		–0.0216	0.2478	0.255	0.034
H(9)	2*i*		0.2704	0.1623	0.332	0.038
H(10)	2*i*		1.6321	0.4869	0.2789	0.048
H(11)	2*i*		1.4432	0.5234	0.26	0.045
H(13)	2*i*		1.6064	0.767	0.4627	0.044
H(14)	2*i*		1.7922	0.726	0.4757	0.047
H(15)	2*i*		1.3833	0.7368	0.3952	0.039
H(16)	2*i*		1.2686	0.5714	0.254	0.04
H(18)	2*i*		1.0471	0.5443	0.1834	0.048
H(19)	2*i*		0.8647	0.5897	0.1693	0.052
H(20)	2*i*		1.0215	0.8216	0.3724	0.047
H(21)	2*i*		1.2079	0.7835	0.3902	0.047
H(22)	2*i*		2.066	0.4235	0.3596	0.045
H(23)	2*i*		2.2649	0.4087	0.3958	0.04
H(25)	2*i*		2.5306	0.586	0.5672	0.035
H(26)	2*i*		2.3664	0.6689	0.5798	0.041
H(27)	2*i*		2.1648	0.6791	0.5373	0.042
H(28)	2*i*		0.4845	0.674	0.1282	0.053
H(29)	2*i*		0.2949	0.7083	0.1046	0.051
H(31)	2*i*		0.4382	0.9456	0.3128	0.058
H(32)	2*i*		0.6255	0.9086	0.3293	0.059
H(33)	2*i*		0.2179	0.9132	0.2398	0.049
H(34)	2*i*		0.1152	0.7526	0.0951	0.044
H(36)	2*i*		0.0408	0.957	0.2347	0.05
H(37)	2*i*		–0.1466	0.993	0.2135	0.054
H(38)	2*i*		–0.2888	0.7677	0.0062	0.054
H(39)	2*i*		–0.1041	0.7256	0.0207	0.05
H(40)	2*i*		–1.0262	0.9193	–0.0719	0.04
H(42)	2*i*		–0.772	1.0797	0.1119	0.054
H(43)	2*i*		–0.5774	1.0599	0.1471	0.057
H(44)	2*i*		–0.6626	0.825	–0.0505	0.05
H(45)	2*i*		–0.8614	0.84	–0.0918	0.046
H(1WA)	2*i*		0.7589	0.072	0.4891	0.091
H(1WB)	2*i*		0.7941	0.1128	0.424	0.091
H(2WA)	2*i*		0.508	0.1468	0.3918	0.086
H(2WB)	2*i*		0.56	0.1029	0.4353	0.086
H(3WA)	2*i*		0.315	0.0505	0.4478	0.11
H(3WB)	2*i*		0.1824	0.0264	0.408	0.11
H(4WA)	2*i*		0.5503	0.3087	0.1173	0.14
H(4WB)	2*i*		0.5393	0.3704	0.0776	0.14
H(5WB)	2*i*	0.5	0.3937	0.4838	0.0902	0.158
H(5WA)	2*i*	0.5	0.5442	0.5053	0.0672	0.158
H(6WA)	2*i*	0.5	0.1226	0.5158	–0.0096	0.186
H(6WB)	2*i*	0.5	0.1261	0.4465	0.0467	0.186
H(7WA)	2*i*		0.1276	0.6331	0.919	0.114
H(7WB)	2*i*		0.2578	0.6403	0.946	0.114
H(8WA)	2*i*		0.8643	0.9376	0.4438	0.087
H(8WB)	2*i*		0.9255	0.9744	0.3893	0.087
H(9WA)	2*i*		0.6344	0.1325	0.2404	0.112
H(9WB)	2*i*		0.7494	0.1129	0.2632	0.112
H(10A)	2*i*		1.8729	0.3758	0.2469	0.096
H(10B)	2*i*		1.7407	0.3759	0.2673	0.096

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	2*i*		0.75523(3)	0.75705(3)	0.25539(2)	0.0292(2)	0.0694(3)	0.0652(3)	0.0314(2)	0.0138(2)	0.0401(2)
Ag(2)	2*i*		1.90390(3)	0.55901(3)	0.39784(3)	0.0267(2)	0.0642(3)	0.0559(3)	0.0275(2)	0.0091(2)	0.0319(2)
Ag(3)	2*i*		–0.41132(4)	0.92640(4)	0.09217(3)	0.0313(2)	0.0992(4)	0.0907(4)	0.0388(2)	0.0186(2)	0.0652(3)
C(1)	2*i*		0.4701(4)	0.2419(3)	0.2596(3)	0.025(2)	0.040(3)	0.032(2)	0.012(2)	–0.001(2)	0.005(2)
C(2)	2*i*		0.1065(4)	0.3531(3)	0.1655(3)	0.042(3)	0.030(2)	0.025(2)	0.017(2)	0.001(2)	0.004(2)
C(3)	2*i*		0.0168(4)	0.1468(3)	0.3534(3)	0.030(2)	0.039(3)	0.038(3)	0.008(2)	0.002(2)	0.015(2)
C(4)	2*i*		0.3285(4)	0.2457(3)	0.2605(3)	0.026(2)	0.029(2)	0.028(2)	0.008(2)	0.001(2)	0.004(2)
C(5)	2*i*		0.2827(4)	0.2952(3)	0.2164(3)	0.030(2)	0.027(2)	0.028(2)	0.010(2)	0.002(2)	0.004(2)
C(6)	2*i*		0.1514(4)	0.2964(3)	0.2137(3)	0.029(2)	0.025(2)	0.027(2)	0.010(2)	–0.001(2)	0.005(2)
C(7)	2*i*		0.0664(4)	0.2478(3)	0.2568(3)	0.022(2)	0.030(2)	0.031(2)	0.012(2)	–0.001(2)	0.006(2)
C(8)	2*i*		0.1101(4)	0.1983(3)	0.3034(3)	0.026(2)	0.028(2)	0.033(2)	0.007(2)	0.001(2)	0.005(2)
C(9)	2*i*		0.2411(4)	0.1970(3)	0.3029(3)	0.033(2)	0.031(2)	0.032(2)	0.015(2)	–0.002(2)	0.009(2)
C(10)	2*i*		1.6264(5)	0.5438(4)	0.3167(3)	0.033(3)	0.044(3)	0.045(3)	0.021(2)	0.004(2)	0.011(2)
C(11)	2*i*		1.5122(5)	0.5653(3)	0.3049(3)	0.031(2)	0.045(3)	0.042(3)	0.022(2)	0.001(2)	0.013(2)
C(12)	2*i*		1.4988(4)	0.6506(3)	0.3603(3)	0.028(2)	0.034(2)	0.036(3)	0.014(2)	0.009(2)	0.018(2)
C(13)	2*i*		1.6090(5)	0.7093(3)	0.4245(3)	0.032(2)	0.038(3)	0.040(3)	0.016(2)	0.002(2)	0.008(2)
C(14)	2*i*		1.7205(5)	0.6840(4)	0.4325(3)	0.033(3)	0.041(3)	0.040(3)	0.015(2)	–0.001(2)	0.008(2)
C(15)	2*i*		1.3818(4)	0.6793(3)	0.3545(3)	0.028(2)	0.038(3)	0.037(3)	0.018(2)	0.007(2)	0.014(2)
C(16)	2*i*		1.2703(4)	0.6291(3)	0.2945(3)	0.031(2)	0.037(2)	0.041(3)	0.020(2)	0.007(2)	0.016(2)
C(17)	2*i*		1.1528(4)	0.6582(3)	0.2883(3)	0.027(2)	0.041(3)	0.040(3)	0.017(2)	0.007(2)	0.019(2)
C(18)	2*i*		1.0446(5)	0.6017(3)	0.2225(3)	0.033(3)	0.037(3)	0.048(3)	0.018(2)	–0.001(2)	0.007(2)
C(19)	2*i*		0.9347(5)	0.6292(4)	0.2143(3)	0.033(3)	0.043(3)	0.050(3)	0.014(2)	–0.003(2)	0.012(2)
C(20)	2*i*		1.0273(5)	0.7654(4)	0.3334(3)	0.035(3)	0.045(3)	0.042(3)	0.025(2)	0.007(2)	0.012(2)
C(21)	2*i*		1.1393(5)	0.7428(3)	0.3447(3)	0.032(2)	0.042(3)	0.041(3)	0.021(2)	0.001(2)	0.005(2)
C(22)	2*i*		2.1281(4)	0.4743(3)	0.4040(3)	0.025(2)	0.038(3)	0.042(3)	0.014(2)	–0.007(2)	0.002(2)
C(23)	2*i*		2.2473(4)	0.4646(3)	0.4256(3)	0.027(2)	0.034(2)	0.036(3)	0.014(2)	–0.004(2)	0.003(2)
C(24)	2*i*		2.3441(4)	0.5385(3)	0.4926(3)	0.022(2)	0.035(2)	0.029(2)	0.011(2)	0.003(2)	0.015(2)
C(25)	2*i*		2.4760(4)	0.5354(3)	0.5196(3)	0.021(2)	0.031(2)	0.030(2)	0.007(2)	–0.004(2)	0.008(2)
C(26)	2*i*		2.3073(4)	0.6183(3)	0.5339(3)	0.030(2)	0.037(3)	0.032(3)	0.016(2)	–0.002(2)	0.006(2)
C(27)	2*i*		2.1850(5)	0.6241(3)	0.5084(3)	0.037(3)	0.032(2)	0.037(3)	0.019(2)	0.004(2)	0.008(2)
C(28)	2*i*		0.4755(5)	0.7299(4)	0.1656(3)	0.041(3)	0.057(3)	0.042(3)	0.029(2)	0.011(2)	0.015(2)
C(29)	2*i*		0.3605(5)	0.7497(4)	0.1516(3)	0.033(3)	0.061(3)	0.035(3)	0.024(2)	0.000(2)	0.013(2)
C(30)	2*i*		0.3401(5)	0.8317(3)	0.2072(3)	0.032(2)	0.047(3)	0.043(3)	0.023(2)	0.010(2)	0.023(2)
C(31)	2*i*		0.4450(5)	0.8898(4)	0.2739(3)	0.036(3)	0.045(3)	0.062(3)	0.022(2)	–0.002(2)	0.008(3)
C(32)	2*i*		0.5576(5)	0.8669(4)	0.2837(4)	0.035(3)	0.050(3)	0.059(3)	0.018(2)	–0.002(2)	0.011(3)
C(33)	2*i*		0.2205(5)	0.8574(4)	0.1985(3)	0.036(3)	0.048(3)	0.046(3)	0.024(2)	0.006(2)	0.017(2)
C(34)	2*i*		0.1135(4)	0.8085(4)	0.1367(3)	0.030(2)	0.047(3)	0.041(3)	0.022(2)	0.007(2)	0.019(2)
C(35)	2*i*		–0.0056(4)	0.8360(3)	0.1296(3)	0.029(2)	0.045(3)	0.040(3)	0.020(2)	0.009(2)	0.023(2)

Table 3. continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(36)	2*i*		–0.0246(5)	0.9172(4)	0.1872(3)	0.031(3)	0.047(3)	0.042(3)	0.021(2)	–0.002(2)	0.004(2)
C(37)	2*i*		–0.1380(5)	0.9382(4)	0.1740(3)	0.038(3)	0.051(3)	0.047(3)	0.026(2)	0.006(2)	0.010(2)
C(38)	2*i*		–0.2212(5)	0.8066(4)	0.0527(3)	0.034(3)	0.053(3)	0.046(3)	0.012(2)	–0.002(2)	0.017(3)
C(39)	2*i*		–0.1101(5)	0.7807(4)	0.0611(3)	0.034(3)	0.041(3)	0.051(3)	0.017(2)	0.000(2)	0.013(2)
C(40)	2*i*		–0.9744(4)	0.9662(3)	–0.0214(3)	0.026(2)	0.033(2)	0.038(3)	0.012(2)	–0.003(2)	0.010(2)
C(41)	2*i*		–0.8447(4)	0.9610(3)	0.0052(3)	0.026(2)	0.031(2)	0.035(3)	0.011(2)	0.001(2)	0.013(2)
C(42)	2*i*		–0.7525(5)	1.0268(4)	0.0775(3)	0.038(3)	0.043(3)	0.047(3)	0.024(2)	–0.008(2)	–0.001(2)
C(43)	2*i*		–0.6355(5)	1.0145(4)	0.0982(3)	0.031(3)	0.052(3)	0.049(3)	0.017(2)	–0.012(2)	0.006(3)
C(44)	2*i*		–0.6847(5)	0.8774(3)	–0.0172(3)	0.038(3)	0.036(3)	0.054(3)	0.021(2)	0.011(2)	0.013(2)
C(45)	2*i*		–0.8054(4)	0.8858(3)	–0.0422(3)	0.031(2)	0.036(3)	0.042(3)	0.014(2)	–0.002(2)	0.005(2)
N(1)	2*i*		2.0938(4)	0.5532(3)	0.4432(2)	0.021(2)	0.039(2)	0.035(2)	0.015(2)	0.004(2)	0.013(2)
N(2)	2*i*		1.7318(4)	0.6006(3)	0.3803(2)	0.024(2)	0.046(2)	0.043(2)	0.019(2)	0.006(2)	0.020(2)
N(3)	2*i*		0.9228(4)	0.7109(3)	0.2685(2)	0.024(2)	0.046(2)	0.048(2)	0.021(2)	0.009(2)	0.023(2)
N(4)	2*i*		0.5770(4)	0.7878(3)	0.2315(2)	0.024(2)	0.048(2)	0.046(2)	0.021(2)	0.006(2)	0.022(2)
N(5)	2*i*		–0.2388(4)	0.8850(3)	0.1077(3)	0.025(2)	0.054(3)	0.054(3)	0.023(2)	0.008(2)	0.031(2)
N(6)	2*i*		–0.5976(4)	0.9407(3)	0.0524(3)	0.022(2)	0.046(2)	0.057(3)	0.018(2)	0.005(2)	0.027(2)
O(1)	2*i*		0.5016(3)	0.1882(2)	0.2940(2)	0.036(2)	0.060(2)	0.052(2)	0.027(2)	0.010(2)	0.026(2)
O(1W)	2*i*		0.7300(4)	0.0994(3)	0.4598(2)	0.057(2)	0.085(3)	0.063(2)	0.039(2)	0.022(2)	0.041(2)
O(2)	2*i*		0.5479(3)	0.2937(3)	0.2260(2)	0.027(2)	0.077(3)	0.083(3)	0.018(2)	0.015(2)	0.048(2)
O(2W)	2*i*		0.4871(4)	0.1210(3)	0.4291(2)	0.053(2)	0.069(2)	0.071(3)	0.033(2)	0.026(2)	0.038(2)
O(3)	2*i*		0.1913(3)	0.4026(3)	0.1342(2)	0.046(2)	0.056(2)	0.056(2)	0.021(2)	0.014(2)	0.034(2)
O(3W)	2*i*		0.2326(4)	0.0228(3)	0.4469(3)	0.049(2)	0.089(3)	0.102(3)	0.021(2)	0.007(2)	0.064(3)
O(4)	2*i*		–0.0156(3)	0.3475(2)	0.1618(2)	0.036(2)	0.057(2)	0.055(2)	0.026(2)	0.003(2)	0.026(2)
O(4W)	2*i*		0.5518(5)	0.3158(4)	0.0694(3)	0.086(3)	0.138(5)	0.081(3)	0.056(3)	0.022(3)	0.053(3)
O(5)	2*i*		–0.0948(3)	0.1576(3)	0.3567(2)	0.034(2)	0.092(3)	0.075(3)	0.029(2)	0.022(2)	0.053(2)
O(5W)	2*i*	0.5	0.446(1)	0.4549(8)	0.0683(6)	0.099(8)	0.140(9)	0.117(8)	0.095(7)	0.026(6)	0.044(7)
O(6)	2*i*		0.0559(3)	0.0959(3)	0.3863(2)	0.040(2)	0.067(2)	0.080(3)	0.021(2)	0.015(2)	0.054(2)
O(6W)	2*i*	0.5	0.115(1)	0.4689(6)	0.0084(5)	0.30(2)	0.052(5)	0.042(5)	0.061(8)	0.026(7)	0.040(4)
O(7W)	2*i*		0.1765(4)	0.6225(3)	0.9537(3)	0.063(3)	0.096(3)	0.073(3)	0.023(2)	–0.009(2)	0.042(3)
O(8W)	2*i*		0.8696(4)	0.9218(3)	0.3910(2)	0.057(2)	0.047(2)	0.063(2)	0.015(2)	0.016(2)	0.014(2)
O(9W)	2*i*		0.6844(4)	0.0988(3)	0.2244(3)	0.059(3)	0.092(3)	0.065(3)	0.044(2)	–0.003(2)	0.000(2)
O(10W)	2*i*		1.8241(4)	0.3887(3)	0.2841(2)	0.038(2)	0.077(3)	0.058(2)	0.016(2)	0.006(2)	0.001(2)

Acknowledgments. We thank the Science Research Foundation for High-level Talents of Pingdingshan University (No.2006046) for support.

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