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catena-Poly[bis[silver(I)-μ-1,2-di-4-pyridylethane-κ^2N:N'] fumarate nonahydrate]

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Key indicators

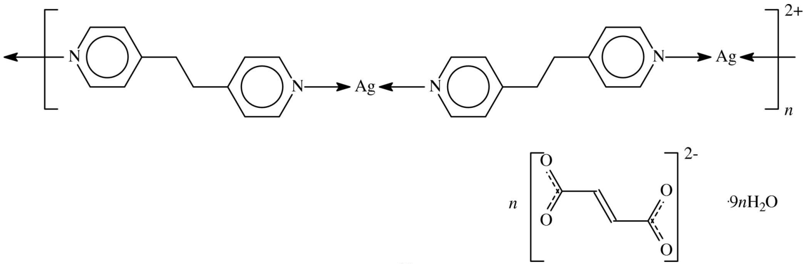
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.035
wR factor = 0.107
Data-to-parameter ratio = 15.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $[Ag_2(C_{12}H_{12}N_2)_2](C_4H_2O_4)\cdot 9H_2O$, the N-heterocycle links adjacent Ag atoms into a polycationic chain; the Ag atom shows linear coordination. The Ag atom lies on a special position of $\overline{1}$ site symmetry, the N-heterocycle about another special position of $\overline{1}$ site symmetry, the dianion about a special position of 2 site symmetry and one water O atom on a special position of 222 site symmetry. The anion and solvent water molecules are hydrogen bonded into a three-dimensional network.

Comment

The 1,2-bis(4-pyridyl)ethane spacer ligand is capable of reacting with silver ions to furnish polycationic chain compounds as found in, for example, the methanesulfonate (Li *et al.*, 2003) and nitrate (LaDuca *et al.*, 2000). A dicarboxylate counter-ion can link the chains into layers; a chain motif has been documented in the 4,4′-biphenyldicarboxylate (Fu *et al.*, 2002). An attempt to synthesize a layer compound with the dianion of malic acid gave the salt (I), with the fumarate dianion, the compound crystallizing with nine water molecules. Oddly, the malate dianion has been transformed to the fumarate dianion, probably by dehydration of the acid during the synthesis.



(I)

The compound adopts a chain motif as the spacer links adjacent Ag atoms into a linear chain; the solvent O1w water molecule is more than 3 Å from atom Ag1 (Fig. 1). Atom Ag1 lies on a special position of $\overline{1}$ site symmetry, the N-heterocycle about another special position of $\overline{1}$ site symmetry, the dianion about a special position of 2.. site symmetry and atom O3w on a special position of 222 site symmetry. The dianion interacts with the solvent water molecules to form a three-dimensional network.

Experimental

1,2-Bis(4-pyridyl)ethane (0.051 g, 0.27 mmol), silver nitrate (0.18 g, 1.06 mmol) and malic acid (0.12 g, 0.90 mmol) were dissolved in a mixture of ethanol (10 ml) and water (5 ml). The solution was refluxed for 5 h. Diethyl ether (20 ml) was layered over the filtered solution in a closed vessel to give single crystals of (I) after a week.

Although malic acid was used as reactant, the product consisted of a fumarate anion. The formation of this anion under the mild reaction conditions was unexpected. Some support for this comes form a report that describes the decomposition of poly(malic acid); heating the polymer gives, among other products, fumaric acid (Fujishige, 2002).

Crystal data

$[Ag_2(C_{12}H_{12}N_2)_2](C_4H_2O_4)\cdot 9H_2O$
$M_r = 860.41$
Orthorhombic, *Fddd*
$a = 13.557$ (1) Å
$b = 15.284$ (1) Å
$c = 33.413$ (3) Å

$V = 6923.3$ (9) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 1.20$ mm^{-1}
$T = 295$ (2) K
$0.45 \times 0.38 \times 0.21$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.586$, $T_{max} = 0.780$

9866 measured reflections
1984 independent reflections
1445 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.107$
$S = 1.11$
1984 reflections
128 parameters

5 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

The dianion is disordered about a twofold rotation axis, and it was refined as a four-atom (O1, O2, C7 and C8) species. The C7—O1 and C7—O2 distances were restrained to 1.250 (5) Å, the C7—C8 distance to 1.500 (5) Å and the C8—C8′ (C8′ is generated by symmetry) to 1.350 (5) Å. The four atoms were restrained to lie on a plane. Somewhat tight restraints were used.

An attempt to set the C8—C8′ distance to 1.50 Å gave a large difference between the unrestrained and restrained goodness of fit, *i.e.* the dianion could not be the succinate dianion.

The carbon-bound H atoms were placed at calculated positions (C—H = 0.93–0.97 Å) and they were included in the refinement in the riding-model approximation with U_{iso}(H) set to $1.2U_{eq}$(C). The water H atoms were placed in calculated positions on the basis of hydrogen bonding but were not refined; U_{iso}(H) = $1.2U_{eq}$(O).

For atom O3*w*, the symmetry-related H31 atom should be generated by the $(\frac{5}{4} - x, y, \frac{1}{4} - z)$ operation.

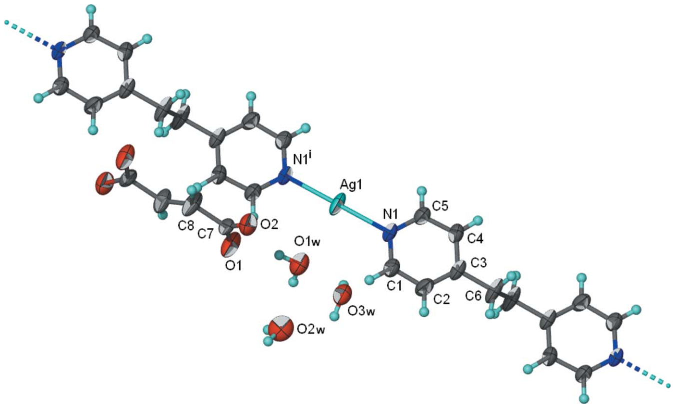


Figure 1

Fig. 1. Molecular strucure of (I). Displacement ellipsoids are drawn at the 50% probability level, and H atoms are shown as spheres of arbitrary radius. [Symmetry code: (i) $\frac{3}{2} - x, 1 - y, \frac{1}{2} - z$.] Unlabelled atoms of the anion are related to labelled atoms by $(-x + \frac{9}{4}, -y + \frac{5}{4}, z)$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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