

Poly[4-(dimethylamino)pyridinium [(μ_6 -5-carboxybenzene-1,2,4-tricarboxyato- κ^6 O¹:O^{1'}:O²:O⁴:O^{4'}:O⁵)diargentate(I)]]

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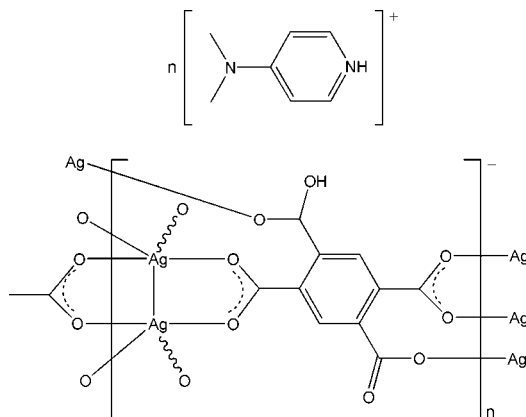
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.018; wR factor = 0.046; data-to-parameter ratio = 11.8.

In the title compound, $\{(\text{C}_7\text{H}_{11}\text{N}_2)[\text{Ag}_2(\text{C}_{10}\text{H}_3\text{O}_8)]\}_n$, the polymeric anion consists of two Ag^{I} atoms and a Hbtc^{3-} ligand (H_4btc = benzene-1,2,4,5-tetracarboxylic acid). Each Ag^{I} atom is coordinated by four O atoms from three different Hbtc^{3-} ligands. The two Ag^{I} atoms are bridged by two bidentate carboxylate groups into an Ag_2O_4 cyclic unit, with an $\text{Ag}\cdots\text{Ag}$ distance of 2.8189 (3) Å. In this way, the Ag atoms are connected by the Hbtc^{3-} ligands into an extended two-dimensional layer structure. A three-dimensional network is accomplished through $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds between the anionic layers. The cationic guest Hdmap^+ [$\text{dmap} = 4$ -(dimethylamino)pyridine] is trapped in the network and adheres to the layer by an $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond.

Related literature

For general background to metal-organic frameworks with 1,2,4,5-benzenetetracarboxylate ligands, see: Cao *et al.* (2002); Hu *et al.* (2004); Li *et al.* (2003). For related complexes, see: Chen (2008); Sun *et al.* (2003); Zheng *et al.* (2002, 2003).



Experimental

Crystal data

$(\text{C}_7\text{H}_{11}\text{N}_2)[\text{Ag}_2(\text{C}_{10}\text{H}_3\text{O}_8)]$
 $M_r = 590.04$
 Triclinic, $P\bar{1}$
 $a = 9.7192$ (3) Å
 $b = 9.9936$ (5) Å
 $c = 10.4968$ (3) Å
 $\alpha = 113.304$ (4)°
 $\beta = 97.140$ (3)°

$\gamma = 103.260$ (3)°
 $V = 884.65$ (7) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 2.27$ mm⁻¹
 $T = 293$ K
 $0.24 \times 0.18 \times 0.14$ mm

Data collection

Oxford Diffraction Gemini R Ultra diffractometer
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
 $T_{\text{min}} = 0.611$, $T_{\text{max}} = 0.725$

7226 measured reflections
 3124 independent reflections
 2808 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.012$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$
 $wR(F^2) = 0.046$
 $S = 1.06$
 3124 reflections

265 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.47$ e Å⁻³

Table 1

Selected bond lengths (Å).

| | | | |
|----------------------|-------------|-----------------------|-------------|
| Ag1—O1 | 2.5220 (15) | Ag2—O4 | 2.2091 (15) |
| Ag1—O3 ⁱ | 2.1784 (15) | Ag2—O5 ⁱⁱⁱ | 2.2224 (16) |
| Ag1—O3 | 2.7573 (19) | Ag2—O5 ^{iv} | 2.873 (2) |
| Ag1—O6 ⁱⁱ | 2.1765 (15) | Ag2—O7 ^{iv} | 2.4442 (15) |

Symmetry codes: (i) $-x + 1, -y + 1, -z + 2$; (ii) $-x + 1, -y, -z + 1$; (iii) $x, y + 1, z + 1$; (iv) $-x + 1, -y, -z + 2$.

Table 2

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{N2}-\text{H}\cdots\text{O7}^v$ | 0.84 | 1.88 | 2.720 (2) | 177 |
| $\text{O2}-\text{H2}\cdots\text{O8}^vi$ | 0.82 | 1.73 | 2.541 (2) | 173 |

Symmetry codes: (v) $x - 1, y + 1, z$; (vi) $x - 1, y, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2204).

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supplementary materials

Acta Cryst. (2009). E65, m994-m995 [doi:10.1107/S1600536809028839]

Poly[4-(dimethylamino)pyridinium [(μ_6 -5-carboxybenzene-1,2,4-tricarboxyato- $\kappa^6 O^1:O^1':O^2:O^4:O^4':O^5$)diargentate(I)]]

X.-F. Zhu, Y.-H. Zhou, L. Guan and H. Zhang

Comment

More efforts have been made to construct MOFs (metal organic frameworks) materials by using 1,2,4,5-benzenetetracarboxylic acid (H_4btc) as molecular building block, owing to its complexed coordination modes to metal ions and various dimensionalities (Cao *et al.*, 2002; Hu *et al.*, 2004; Li *et al.*, 2003). According to literature, the combination of H_4btc , as a polydentate ligand and silver(I) can produce various architectures, involving in $[Ag_2(pbi)_2(H_2btc)]_n$ [$pbi = 2$ -(3-pyridyl)-1*H*-benzimidazole] (Chen, 2008), $[Ag(\mu_3-hmt)]_2[Ag(NH_3)_2](btc).3H_2O$ ($hmt =$ hexamethylenetetramine) (Zheng *et al.*, 2002), $[Ag_8(\mu_3-hmt)_2(\mu_4-hmt)_2(\mu-btc)_2(\mu-H_2O)_3].18H_2O$ (Zheng *et al.*, 2003), and $[Ag(bipy)][H_2btc]_{0.5}.H_2O$ (Sun *et al.*, 2003). Herein, the title complex, $[Hdmap][Ag_2(Hbtc)]$ ($dmap = 4$ -dimethylaminopyridine), with a layer structure is reported.

The structure of the title compound contains two crystallographically independent Ag^I atoms, one $(Hbtc)^{3-}$ ligand and one $(Hdmap)^+$ cation. Each Ag^I atom is coordinated by four carboxylate O atoms from three different $Hbtc$ ligands (Fig. 1), with three close bond distances [average $Ag1-O = 2.2923$ (15) and $Ag2-O = 2.2919$ (15) Å] and one long bond distance [$Ag1-O = 2.7573$ (19) and $Ag2-O = 2.873$ (2) Å] (Table 1). It is worth noting that two adjacent $Ag1$ and $Ag2$ atoms are bridged by two bidentate carboxylate groups into an Ag_2O_4 cyclic unit, with an $Ag\cdots Ag$ distance of 2.8189 (3) Å. The $Hbtc$ ligand connects six Ag atoms, leading to a two-dimensional anionic layer (Fig. 2). The interlayer $O-H\cdots O$ hydrogen bonds hold adjacent layers together to bring out a supramolecular network. The cationic guest $(Hdmap)^+$ is trapped in the network and adhere to the layer by an $N-H\cdots O$ hydrogen bond (Table 2).

Experimental

A mixture of pyromelic acid anhydride (0.218 g, 0.1 mmol) in distilled water (10 ml) was stirred at 333 K for 1 h until to get clear solution and then a DMF solution (2 ml) of $AgNO_3$ (0.169 g, 0.1 mmol) was added on stirring for 1 h under ambient condition. The resulting solution was allowed to stand in air at room temperature for 3 d. Colorless crystals were collected in 77.8% yield based on $AgNO_3$.

Refinement

H atoms were positioned geometrically and refined as riding atoms, with $C-H = 0.93$ (aromatic) and 0.96 (methyl), $N-H = 0.84$ and $O-H = 0.82$ Å, and with $U_{iso}(H) = 1.2$ (or 1.5 for methyl and hydroxyl) $U_{eq}(C, N, O)$.

Figures

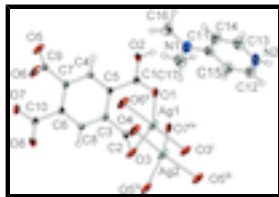


Fig. 1. The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $1 - x, 1 - y, 2 - z$; (ii) $1 - x, -y, 1 - z$; (iii) $x, 1 + y, 1 + z$; (iv) $1 - x, -y, 2 - z$.]

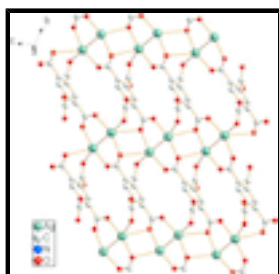


Fig. 2. A view of the two-dimensional layer in the title compound. H atoms are omitted for clarity.

Poly[4-(dimethylamino)pyridinium [(μ_6 -5-carboxybenzene-1,2,4-tricarboxylato- $\kappa^6 O^1:O^1':O^2:O^4:O^4':O^5$)diargentate(I)]]

Crystal data

(C₇H₁₁N₂)[Ag₂(C₁₀H₃O₈)]

$M_r = 590.04$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 9.7192$ (3) Å

$b = 9.9936$ (5) Å

$c = 10.4968$ (3) Å

$\alpha = 113.304$ (4)°

$\beta = 97.140$ (3)°

$\gamma = 103.260$ (3)°

$V = 884.65$ (7) Å³

$Z = 2$

$F_{000} = 576$

$D_x = 2.215$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3901 reflections

$\theta = 4.4$ – 25.0 °

$\mu = 2.27$ mm⁻¹

$T = 293$ K

Block, colorless

$0.24 \times 0.18 \times 0.14$ mm

Data collection

Oxford Diffraction Gemini R Ultra diffractometer

3124 independent reflections

Radiation source: fine-focus sealed tube

2808 reflections with $I > 2\sigma(I)$

Monochromator: graphite

$R_{int} = 0.012$

Detector resolution: 10.0 pixels mm⁻¹

$\theta_{max} = 25.0$ °

$T = 293$ K

$\theta_{min} = 4.4$ °

ω scans

$h = -11 \rightarrow 11$

Absorption correction: multi-scan

$k = -11 \rightarrow 11$

(CrysAlis RED; Oxford Diffraction, 2006)

$T_{min} = 0.611$, $T_{max} = 0.725$

$l = -12 \rightarrow 12$

7226 measured reflections

Refinement

| | |
|--|---|
| Refinement on F^2 | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.018$ | $w = 1/[\sigma^2(F_o^2) + (0.0251P)^2 + 0.4969P]$ |
| $wR(F^2) = 0.046$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.06$ | $(\Delta/\sigma)_{\max} = 0.003$ |
| 3124 reflections | $\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$ |
| 265 parameters | $\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Secondary atom site location: difference Fourier map | Extinction coefficient: 0.0157 (6) |

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Ag1 | 0.421397 (19) | 0.410977 (18) | 0.802682 (17) | 0.02690 (8) |
| Ag2 | 0.421385 (19) | 0.385653 (19) | 1.290598 (17) | 0.02889 (9) |
| O1 | 0.22857 (16) | 0.20334 (17) | 0.81649 (18) | 0.0277 (4) |
| O2 | 0.10698 (15) | -0.04467 (17) | 0.71974 (19) | 0.0329 (4) |
| H2 | 0.0364 | -0.0126 | 0.7220 | 0.049* |
| O3 | 0.57621 (18) | 0.37804 (17) | 1.02125 (17) | 0.0326 (4) |
| O4 | 0.4252 (2) | 0.21751 (18) | 1.07819 (17) | 0.0344 (4) |
| O5 | 0.41174 (19) | -0.39642 (18) | 0.45762 (18) | 0.0357 (4) |
| O6 | 0.5636 (2) | -0.23946 (18) | 0.39821 (17) | 0.0341 (4) |
| O7 | 0.75751 (16) | -0.21312 (17) | 0.65497 (17) | 0.0251 (3) |
| O8 | 0.87465 (16) | 0.03113 (17) | 0.7214 (2) | 0.0336 (4) |
| C1 | 0.2265 (2) | 0.0702 (2) | 0.7685 (2) | 0.0178 (4) |
| C2 | 0.4977 (2) | 0.2487 (2) | 0.9985 (2) | 0.0173 (4) |
| C3 | 0.4930 (2) | 0.1181 (2) | 0.8600 (2) | 0.0160 (4) |
| C4 | 0.3628 (2) | -0.0996 (2) | 0.6355 (2) | 0.0177 (4) |
| H4 | 0.2755 | -0.1617 | 0.5684 | 0.021* |
| C5 | 0.3627 (2) | 0.0251 (2) | 0.7570 (2) | 0.0160 (4) |
| C6 | 0.6223 (2) | -0.0377 (2) | 0.7128 (2) | 0.0157 (4) |
| C7 | 0.4913 (2) | -0.1332 (2) | 0.6121 (2) | 0.0153 (4) |
| C8 | 0.6218 (2) | 0.0862 (2) | 0.8348 (2) | 0.0185 (4) |
| H8 | 0.7093 | 0.1493 | 0.9012 | 0.022* |
| C9 | 0.4888 (2) | -0.2673 (2) | 0.4775 (2) | 0.0168 (4) |
| C10 | 0.7617 (2) | -0.0765 (2) | 0.6939 (2) | 0.0192 (4) |
| N1 | 0.0720 (2) | 0.4030 (2) | 0.1392 (2) | 0.0333 (5) |
| N2 | -0.1309 (2) | 0.6318 (3) | 0.4369 (2) | 0.0378 (5) |
| H | -0.1683 | 0.6777 | 0.5021 | 0.045* |
| C11 | 0.0048 (2) | 0.4765 (3) | 0.2354 (2) | 0.0271 (5) |
| C12 | -0.0362 (3) | 0.7091 (3) | 0.3890 (3) | 0.0354 (6) |

supplementary materials

| | | | | |
|------|-------------|------------|------------|------------|
| H12 | -0.0172 | 0.8144 | 0.4240 | 0.042* |
| C13 | -0.1624 (3) | 0.4795 (3) | 0.3866 (3) | 0.0375 (6) |
| H13 | -0.2296 | 0.4279 | 0.4204 | 0.045* |
| C14 | -0.0985 (3) | 0.3988 (3) | 0.2872 (3) | 0.0343 (6) |
| H14 | -0.1223 | 0.2931 | 0.2533 | 0.041* |
| C15 | 0.0325 (3) | 0.6369 (3) | 0.2906 (3) | 0.0333 (6) |
| H15 | 0.0981 | 0.6930 | 0.2592 | 0.040* |
| C16 | 0.0426 (3) | 0.2378 (3) | 0.0780 (3) | 0.0407 (6) |
| H16A | -0.0540 | 0.1883 | 0.0172 | 0.061* |
| H16B | 0.0505 | 0.2065 | 0.1535 | 0.061* |
| H16C | 0.1119 | 0.2095 | 0.0231 | 0.061* |
| C17 | 0.1797 (3) | 0.4867 (4) | 0.0902 (3) | 0.0423 (7) |
| H17A | 0.2588 | 0.5591 | 0.1700 | 0.063* |
| H17B | 0.1351 | 0.5400 | 0.0473 | 0.063* |
| H17C | 0.2160 | 0.4161 | 0.0210 | 0.063* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|-------------|-------------|-------------|
| Ag1 | 0.03846 (13) | 0.01606 (11) | 0.01917 (11) | 0.00464 (7) | 0.01347 (8) | 0.00093 (7) |
| Ag2 | 0.04268 (13) | 0.01682 (11) | 0.02185 (11) | 0.00567 (8) | 0.01695 (8) | 0.00245 (8) |
| O1 | 0.0209 (8) | 0.0163 (8) | 0.0436 (10) | 0.0084 (6) | 0.0142 (7) | 0.0076 (7) |
| O2 | 0.0123 (7) | 0.0184 (8) | 0.0581 (11) | 0.0053 (6) | 0.0090 (7) | 0.0065 (8) |
| O3 | 0.0409 (10) | 0.0154 (8) | 0.0288 (9) | 0.0006 (7) | 0.0191 (7) | -0.0016 (7) |
| O4 | 0.0535 (11) | 0.0211 (8) | 0.0240 (8) | 0.0075 (7) | 0.0231 (8) | 0.0037 (7) |
| O5 | 0.0431 (10) | 0.0148 (8) | 0.0356 (9) | -0.0001 (7) | 0.0255 (8) | -0.0019 (7) |
| O6 | 0.0553 (11) | 0.0201 (8) | 0.0233 (8) | 0.0081 (7) | 0.0245 (8) | 0.0034 (7) |
| O7 | 0.0233 (8) | 0.0173 (8) | 0.0361 (9) | 0.0103 (6) | 0.0155 (7) | 0.0084 (7) |
| O8 | 0.0143 (8) | 0.0191 (8) | 0.0614 (12) | 0.0046 (6) | 0.0133 (7) | 0.0108 (8) |
| C1 | 0.0168 (10) | 0.0168 (11) | 0.0191 (10) | 0.0056 (8) | 0.0084 (8) | 0.0058 (9) |
| C2 | 0.0181 (10) | 0.0166 (10) | 0.0149 (9) | 0.0087 (8) | 0.0047 (8) | 0.0027 (8) |
| C3 | 0.0185 (10) | 0.0132 (10) | 0.0154 (10) | 0.0056 (8) | 0.0061 (8) | 0.0045 (8) |
| C4 | 0.0146 (10) | 0.0155 (10) | 0.0173 (10) | 0.0036 (8) | 0.0026 (8) | 0.0026 (8) |
| C5 | 0.0151 (10) | 0.0132 (10) | 0.0199 (10) | 0.0049 (7) | 0.0079 (8) | 0.0061 (8) |
| C6 | 0.0144 (10) | 0.0132 (10) | 0.0185 (10) | 0.0047 (7) | 0.0067 (8) | 0.0050 (8) |
| C7 | 0.0173 (10) | 0.0130 (9) | 0.0160 (9) | 0.0063 (8) | 0.0073 (8) | 0.0049 (8) |
| C8 | 0.0155 (10) | 0.0143 (10) | 0.0187 (10) | 0.0028 (8) | 0.0031 (8) | 0.0016 (8) |
| C9 | 0.0168 (10) | 0.0164 (10) | 0.0150 (10) | 0.0071 (8) | 0.0035 (8) | 0.0036 (8) |
| C10 | 0.0181 (10) | 0.0173 (11) | 0.0199 (10) | 0.0063 (8) | 0.0074 (8) | 0.0045 (9) |
| N1 | 0.0372 (11) | 0.0354 (11) | 0.0322 (11) | 0.0154 (9) | 0.0194 (9) | 0.0141 (9) |
| N2 | 0.0413 (12) | 0.0458 (13) | 0.0318 (11) | 0.0272 (10) | 0.0174 (10) | 0.0123 (10) |
| C11 | 0.0270 (12) | 0.0322 (13) | 0.0235 (11) | 0.0108 (10) | 0.0076 (9) | 0.0122 (10) |
| C12 | 0.0420 (14) | 0.0289 (13) | 0.0338 (13) | 0.0143 (11) | 0.0061 (11) | 0.0113 (11) |
| C13 | 0.0364 (14) | 0.0422 (15) | 0.0414 (14) | 0.0136 (11) | 0.0216 (12) | 0.0213 (13) |
| C14 | 0.0383 (14) | 0.0280 (13) | 0.0401 (14) | 0.0111 (11) | 0.0188 (12) | 0.0150 (12) |
| C15 | 0.0369 (13) | 0.0308 (13) | 0.0344 (13) | 0.0097 (10) | 0.0128 (11) | 0.0156 (11) |
| C16 | 0.0437 (15) | 0.0410 (16) | 0.0369 (14) | 0.0218 (12) | 0.0152 (12) | 0.0102 (13) |
| C17 | 0.0371 (14) | 0.0576 (18) | 0.0399 (15) | 0.0176 (13) | 0.0225 (12) | 0.0236 (14) |

Geometric parameters (Å, °)

| | | | |
|---|-------------|------------|-------------|
| Ag1—O1 | 2.5220 (15) | C6—C8 | 1.387 (3) |
| Ag1—O3 ⁱ | 2.1784 (15) | C6—C7 | 1.400 (3) |
| Ag1—O3 | 2.7573 (19) | C6—C10 | 1.507 (3) |
| Ag1—O6 ⁱⁱ | 2.1765 (15) | C7—C9 | 1.507 (3) |
| Ag2—O4 | 2.2091 (15) | C8—H8 | 0.9300 |
| Ag2—O5 ⁱⁱⁱ | 2.2224 (16) | N1—C11 | 1.336 (3) |
| Ag2—O5 ^{iv} | 2.873 (2) | N1—C16 | 1.455 (3) |
| Ag2—O7 ^{iv} | 2.4442 (15) | N1—C17 | 1.460 (3) |
| Ag1—Ag2 ⁱ | 2.8189 (3) | N2—C12 | 1.339 (3) |
| O1—C1 | 1.216 (3) | N2—C13 | 1.341 (3) |
| O2—C1 | 1.306 (2) | N2—H | 0.84 |
| O2—H2 | 0.82 | C11—C15 | 1.417 (3) |
| O3—C2 | 1.252 (3) | C11—C14 | 1.420 (3) |
| O4—C2 | 1.244 (3) | C12—C15 | 1.355 (3) |
| O5—C9 | 1.255 (3) | C12—H12 | 0.9300 |
| O6—C9 | 1.240 (3) | C13—C14 | 1.361 (3) |
| O7—C10 | 1.249 (3) | C13—H13 | 0.9300 |
| O8—C10 | 1.257 (2) | C14—H14 | 0.9300 |
| C1—C5 | 1.496 (3) | C15—H15 | 0.9300 |
| C2—C3 | 1.508 (3) | C16—H16A | 0.9600 |
| C3—C8 | 1.392 (3) | C16—H16B | 0.9600 |
| C3—C5 | 1.401 (3) | C16—H16C | 0.9600 |
| C4—C5 | 1.387 (3) | C17—H17A | 0.9600 |
| C4—C7 | 1.392 (3) | C17—H17B | 0.9600 |
| C4—H4 | 0.9300 | C17—H17C | 0.9600 |
| O6 ⁱⁱ —Ag1—O3 ⁱ | 165.22 (6) | C4—C7—C6 | 118.95 (18) |
| O6 ⁱⁱ —Ag1—O1 | 88.08 (6) | C4—C7—C9 | 120.01 (17) |
| O3 ⁱ —Ag1—O1 | 104.84 (6) | C6—C7—C9 | 120.99 (17) |
| O6 ⁱⁱ —Ag1—Ag2 ⁱ | 82.64 (4) | C6—C8—C3 | 121.33 (18) |
| O3 ⁱ —Ag1—Ag2 ⁱ | 83.04 (4) | C6—C8—H8 | 119.3 |
| O1—Ag1—Ag2 ⁱ | 162.98 (4) | C3—C8—H8 | 119.3 |
| O3—Ag1—O1 | 80.23 (5) | O6—C9—O5 | 126.56 (19) |
| O3—Ag1—O3 ⁱ | 82.06 (6) | O6—C9—C7 | 117.07 (18) |
| O3—Ag1—O6 ⁱⁱ | 107.63 (5) | O5—C9—C7 | 116.36 (17) |
| O3—Ag1—Ag2 ⁱ | 116.16 (4) | O7—C10—O8 | 124.57 (19) |
| O4—Ag2—O5 ⁱⁱⁱ | 158.69 (7) | O7—C10—C6 | 117.63 (17) |
| O4—Ag2—O7 ^{iv} | 97.68 (6) | O8—C10—C6 | 117.78 (18) |
| O5 ⁱⁱⁱ —Ag2—O7 ^{iv} | 97.08 (5) | C11—N1—C16 | 122.5 (2) |
| O4—Ag2—Ag1 ⁱ | 81.08 (4) | C11—N1—C17 | 120.8 (2) |
| O5 ⁱⁱⁱ —Ag2—Ag1 ⁱ | 81.40 (4) | C16—N1—C17 | 116.8 (2) |
| O7 ^{iv} —Ag2—Ag1 ⁱ | 168.58 (4) | C12—N2—C13 | 120.7 (2) |

supplementary materials

| | | | |
|---|--------------|------------------------------|--------------|
| O5 ^{iv} —Ag2—O4 | 119.23 (5) | C12—N2—H | 120.9 |
| O5 ^{iv} —Ag2—O5 ⁱⁱⁱ | 78.86 (6) | C13—N2—H | 118.3 |
| O5 ^{iv} —Ag2—O7 ^{iv} | 78.31 (5) | N1—C11—C15 | 121.4 (2) |
| O5 ^{iv} —Ag2—Ag1 ⁱ | 112.29 (4) | N1—C11—C14 | 122.1 (2) |
| C1—O1—Ag1 | 123.41 (13) | C15—C11—C14 | 116.4 (2) |
| C1—O2—H2 | 109.5 | N2—C12—C15 | 121.3 (2) |
| C2—O3—Ag1 ⁱ | 123.07 (13) | N2—C12—H12 | 119.3 |
| C2—O4—Ag2 | 124.47 (14) | C15—C12—H12 | 119.3 |
| C9—O5—Ag2 ^v | 123.12 (13) | N2—C13—C14 | 121.5 (2) |
| C9—O6—Ag1 ⁱⁱ | 124.62 (14) | N2—C13—H13 | 119.3 |
| C10—O7—Ag2 ^{iv} | 121.06 (12) | C14—C13—H13 | 119.3 |
| O1—C1—O2 | 123.50 (18) | C13—C14—C11 | 119.7 (2) |
| O1—C1—C5 | 122.02 (17) | C13—C14—H14 | 120.1 |
| O2—C1—C5 | 114.46 (17) | C11—C14—H14 | 120.1 |
| O4—C2—O3 | 126.86 (19) | C12—C15—C11 | 120.3 (2) |
| O4—C2—C3 | 117.34 (18) | C12—C15—H15 | 119.8 |
| O3—C2—C3 | 115.79 (17) | C11—C15—H15 | 119.8 |
| C8—C3—C5 | 118.74 (18) | N1—C16—H16A | 109.5 |
| C8—C3—C2 | 119.06 (17) | N1—C16—H16B | 109.5 |
| C5—C3—C2 | 122.18 (17) | H16A—C16—H16B | 109.5 |
| C5—C4—C7 | 121.14 (18) | N1—C16—H16C | 109.5 |
| C5—C4—H4 | 119.4 | H16A—C16—H16C | 109.5 |
| C7—C4—H4 | 119.4 | H16B—C16—H16C | 109.5 |
| C4—C5—C3 | 119.97 (18) | N1—C17—H17A | 109.5 |
| C4—C5—C1 | 119.38 (17) | N1—C17—H17B | 109.5 |
| C3—C5—C1 | 120.24 (17) | H17A—C17—H17B | 109.5 |
| C8—C6—C7 | 119.81 (18) | N1—C17—H17C | 109.5 |
| C8—C6—C10 | 120.10 (17) | H17A—C17—H17C | 109.5 |
| C7—C6—C10 | 119.93 (17) | H17B—C17—H17C | 109.5 |
| O6 ⁱⁱ —Ag1—O1—C1 | 31.61 (18) | C10—C6—C7—C9 | -5.6 (3) |
| O3 ⁱ —Ag1—O1—C1 | -155.65 (17) | C7—C6—C8—C3 | 0.1 (3) |
| Ag2 ⁱ —Ag1—O1—C1 | 88.4 (2) | C10—C6—C8—C3 | -175.26 (19) |
| O5 ⁱⁱⁱ —Ag2—O4—C2 | 43.7 (3) | C5—C3—C8—C6 | -2.1 (3) |
| O7 ^{iv} —Ag2—O4—C2 | 177.18 (18) | C2—C3—C8—C6 | 176.43 (19) |
| Ag1 ⁱ —Ag2—O4—C2 | 8.66 (17) | Ag1 ⁱⁱ —O6—C9—O5 | 1.1 (3) |
| Ag1—O1—C1—O2 | -150.04 (16) | Ag1 ⁱⁱ —O6—C9—C7 | -179.62 (13) |
| Ag1—O1—C1—C5 | 28.3 (3) | Ag2 ^v —O5—C9—O6 | 11.0 (3) |
| Ag2—O4—C2—O3 | -2.3 (3) | Ag2 ^v —O5—C9—C7 | -168.32 (13) |
| Ag2—O4—C2—C3 | 177.08 (13) | C4—C7—C9—O6 | 120.2 (2) |
| Ag1 ⁱ —O3—C2—O4 | -9.4 (3) | C6—C7—C9—O6 | -57.5 (3) |
| Ag1 ⁱ —O3—C2—C3 | 171.21 (13) | C4—C7—C9—O5 | -60.4 (3) |
| O4—C2—C3—C8 | -119.5 (2) | C6—C7—C9—O5 | 121.9 (2) |
| O3—C2—C3—C8 | 60.0 (3) | Ag2 ^{iv} —O7—C10—O8 | 151.16 (17) |
| O4—C2—C3—C5 | 59.0 (3) | Ag2 ^{iv} —O7—C10—C6 | -27.2 (2) |

| | | | |
|--------------|--------------|-----------------|------------|
| O3—C2—C3—C5 | -121.6 (2) | C8—C6—C10—O7 | 132.8 (2) |
| C7—C4—C5—C3 | -1.1 (3) | C7—C6—C10—O7 | -42.6 (3) |
| C7—C4—C5—C1 | 171.50 (18) | C8—C6—C10—O8 | -45.6 (3) |
| C8—C3—C5—C4 | 2.6 (3) | C7—C6—C10—O8 | 139.0 (2) |
| C2—C3—C5—C4 | -175.89 (19) | C16—N1—C11—C15 | -178.3 (2) |
| C8—C3—C5—C1 | -170.00 (18) | C17—N1—C11—C15 | 1.6 (3) |
| C2—C3—C5—C1 | 11.5 (3) | C16—N1—C11—C14 | 1.2 (4) |
| O1—C1—C5—C4 | -138.5 (2) | C17—N1—C11—C14 | -178.8 (2) |
| O2—C1—C5—C4 | 39.9 (3) | C13—N2—C12—C15 | -1.2 (4) |
| O1—C1—C5—C3 | 34.1 (3) | C12—N2—C13—C14 | 0.9 (4) |
| O2—C1—C5—C3 | -147.48 (19) | N2—C13—C14—C11 | 0.3 (4) |
| C5—C4—C7—C6 | -0.8 (3) | N1—C11—C14—C13 | 179.2 (2) |
| C5—C4—C7—C9 | -178.56 (19) | C15—C11—C14—C13 | -1.2 (3) |
| C8—C6—C7—C4 | 1.3 (3) | N2—C12—C15—C11 | 0.2 (4) |
| C10—C6—C7—C4 | 176.73 (18) | N1—C11—C15—C12 | -179.5 (2) |
| C8—C6—C7—C9 | 179.04 (19) | C14—C11—C15—C12 | 0.9 (3) |

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x+1, -y, -z+1$; (iii) $x, y+1, z+1$; (iv) $-x+1, -y, -z+2$; (v) $x, y-1, z-1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N2—H \cdots O7 ^{vi} | 0.84 | 1.88 | 2.720 (2) | 177 |
| O2—H2 \cdots O8 ^{vii} | 0.82 | 1.73 | 2.541 (2) | 173 |

Symmetry codes: (vi) $x-1, y+1, z$; (vii) $x-1, y, z$.

Fig. 1

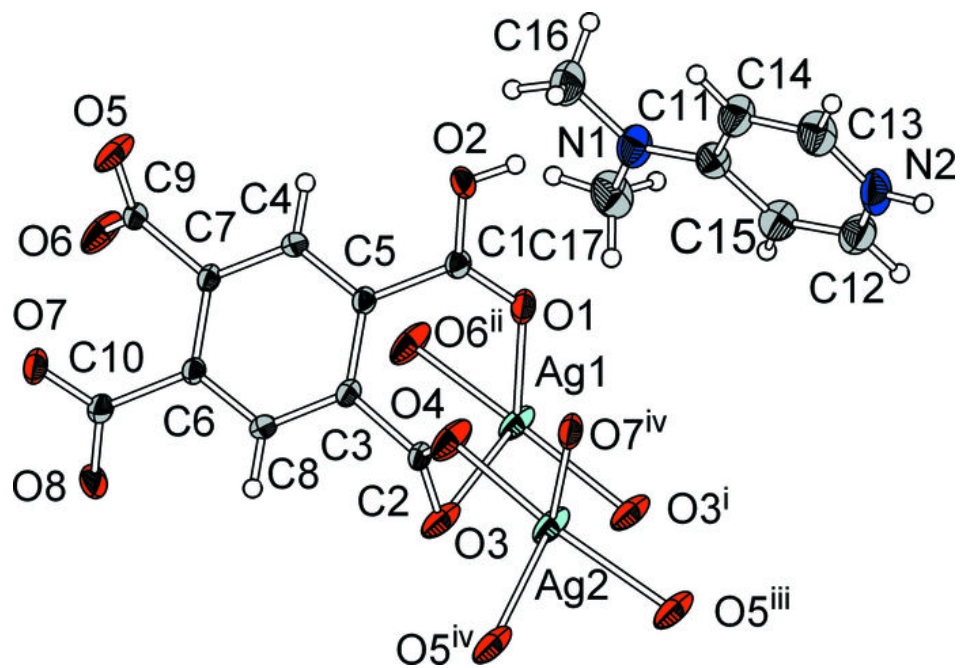


Fig. 2

