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Bis[μ-2-(3-pyridyl)benzimidazole]bis-[bis(3,5-dicarboxybenzoato)silver(I)]

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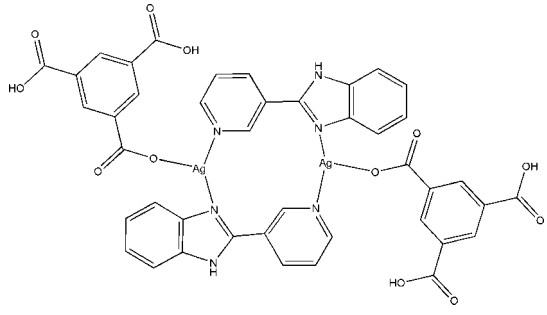
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.070; data-to-parameter ratio = 14.2.

In the binuclear title complex, $[Ag_2(C_9H_5O_6)_2(C_{12}H_9N_3)_2]$, the two Ag^I atoms are in a trigonal planar environment formed by two N atoms from two 2-(3-pyridyl)benzimidazole ligands and one O atom from a 3,5-dicarboxybenzoate ligand, forming a centrosymmetric cyclic dimer. Intermolecular N—H···O and O—H···O hydrogen-bonding interactions aggregate the binuclear units into a three-dimensional framework.

Related literature

For related literature, see: Dai *et al.* (2003); Prior *et al.* (2003); Plater *et al.* (2001); Alcalde *et al.* (1992).



Experimental

Crystal data

$[Ag_2(C_9H_5O_6)_2(C_{12}H_9N_3)_2]$
M_r = 1024.44
Monoclinic, $P2_1/c$
a = 14.573 (3) Å
b = 7.7160 (15) Å
c = 16.662 (4) Å
β = 105.038 (3)°
V = 1809.3 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.16 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2002)
T_{min} = 0.756, T_{max} = 0.881
13327 measured reflections
4111 independent reflections
3895 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.070
S = 1.09
4111 reflections
289 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—N2	2.1508 (18)	Ag1—O2	2.5320 (17)
Ag1—N1^{i}	2.1729 (18)		
N2—Ag1—N1^{i}	152.96 (7)	N1^{i}—Ag1—O2	107.84 (7)
N2—Ag1—O2	99.17 (7)		

Symmetry code: (i) $-x+1, -y+1, -z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···O1^{ii}	0.86	2.01	2.828 (2)	158
O3—H3C···O6^{iii}	0.841 (18)	1.941 (19)	2.772 (2)	169 (4)
O6—H6B···O1^{iv}	0.841 (18)	1.647 (19)	2.487 (2)	176 (4)

Symmetry codes: (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Siemens, 1994); 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: BT2568).

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supplementary materials

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Bis[μ-2-(3-pyridyl)benzimidazole]bis[bis(3,5-dicarboxybenzoato)silver(I)]

C.-K. Xia, W. Wu, L. Qiu and J.-M. Xie

Comment

1,3,5-Benzenetricarboxylatic acid has been often be used to construct supramolecular structures (Dai *et al.*, 2003, Prior *et al.*, 2003; Plater *et al.*, 2001). In those coordination complexes, the 1,3,5-benzenetricarboxylatic acid ligand demonstrated versatile coordination modes and the tendence of forming hydrogen bonds. The structures are controlled by the extent of the depronation of 1,3,5-benzenetricarboxylatic acid, the nature of the auxiliary ligands, and the metal centers.

In the title compound each two Ag^{I} atom is bonded by two nitrogen atoms from two two 2-(3-pyridyl)benzimidazole ligands, forming a binuclear macrocycle. The coordination sphere is completed by a 3,5-dicarboxybenzoate ligand. The intermolecular N—H···O hydrogen bonding interactions connect the binuclear units into a two-dimensional undulating sheet with the protonated carboxylic acid groups on the opposite sides (Fig. 2). The sheets are further connected by O—H···O hydrogen bonding interactions into a three-dimensional framework (Fig. 3). Although there are aromatic rings, no apparent $\pi\cdots\pi$ stacking interactions have been found.

Experimental

The 2-(3-pyridyl)benzimidazole was synthesized according to the literature (Alcalde *et al.*, 1992). A solution of $AgNO_3$ (0.104 g, 0.61 mmol), 2-(3-pyridyl)benzimidazole (0.14 g, 0.61 mmol), 1,3,5-benzenetricarboxylatic acid (0.128 g, 0.61 mmol) and H_2O (15 ml) was stirred under ambient condition, adjust with dilute NaOH to pH=7.3, then sealed in Teflon-lined stainless steel vessel, heated at 403 K for 4 days and cooled to room temperature. The resulting product was recovered by filtration, washed with distilled water and dried in air. (60% yield).

Refinement

After checking their presence in the different map, H atoms bonded to C and N were fixed geometrically and allowed to ride on their attached atoms, with C—H=0.93 Å, N—H=0.86 Å, *U*iso(H)=1.2*U*eq(C,*N*). The two carboxylic acid H atoms were located from a difference map and refined with a restraint of 0.83 (1)Å for the O—H distance.

Figures

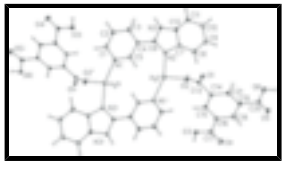

Fig. 1. The structure of (I), with the atomic labels and 30% probability displacement ellipsoids for non-H atoms. [Symmetry code: (i) $1-x, 1-y, -z$.]

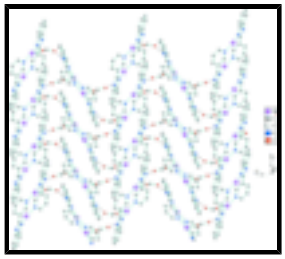

Fig. 2. A view of the two-dimensional sheet of (I). The phenyl ring and two protonated carboxylic acid groups of the 3,5-dicarboxybenzoate ligands are omitted for clearity.

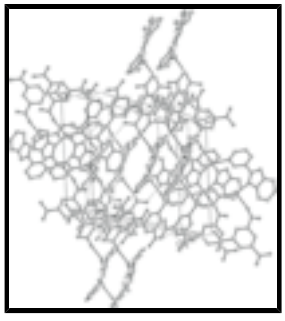

Fig. 3. Three-dimensional supramolecular architecture constructed by intermolecular N—H···O and O—H···O hydrogen bonding interactions.

Bis[μ-2-(3-pyridyl)benzimidazole]bis[bis(3,5-dicarboxybenzoato)silver(I)]

Crystal data

$[Ag_2(C_9H_5O_6)_2(C_{12}H_9N_3)_2]$	F_{000} = 1024
M_r = 1024.44	D_x = 1.880 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -p 2ybc	Cell parameters from 4455 reflections
a = 14.573 (3) Å	θ = 2.2–27.5º
b = 7.7160 (15) Å	μ = 1.16 mm^{-1}
c = 16.662 (4) Å	T = 293 (2) K
β = 105.038 (3)º	Prism, white
V = 1809.3 (7) Å^3	0.30 × 0.20 × 0.10 mm
Z = 2	

Data collection

Rigaku Mercury CCD diffractometer	4111 independent reflections
Radiation source: fine-focus sealed tube	3895 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 293(2) K	θ_{max} = 27.5º
ω scan	θ_{min} = 2.5º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2002)	h = −18→17
T_{min} = 0.756, T_{max} = 0.881	k = −9→9
13327 measured reflections	l = −21→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.070$	$w = 1/[\sigma^2(F_o^2) + (0.0272P)^2 + 1.8597P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
4111 reflections	$\Delta\rho_{max} = 0.57$ e Å^{-3}
289 parameters	$\Delta\rho_{min} = -0.80$ e Å^{-3}
2 restraints	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0084 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.382999 (13)	0.29268 (3)	0.037544 (12)	0.03468 (9)
C1	0.74977 (16)	0.5248 (3)	0.11466 (15)	0.0309 (5)
H1A	0.7874	0.6219	0.1140	0.037*
C2	0.77957 (18)	0.4034 (3)	0.17668 (16)	0.0368 (6)
H2A	0.8373	0.4177	0.2161	0.044*
C3	0.72380 (17)	0.2614 (3)	0.18019 (14)	0.0295 (5)
H3A	0.7427	0.1799	0.2224	0.035*
C4	0.63816 (14)	0.2416 (3)	0.11918 (13)	0.0217 (4)
C5	0.61437 (14)	0.3674 (3)	0.05766 (13)	0.0243 (4)
H5A	0.5583	0.3538	0.0161	0.029*
C6	0.57592 (14)	0.0933 (3)	0.12172 (12)	0.0220 (4)
C7	0.45294 (15)	−0.0690 (3)	0.11505 (13)	0.0236 (4)
C8	0.36146 (16)	−0.1367 (3)	0.10454 (15)	0.0309 (5)
H8A	0.3076	−0.0719	0.0802	0.037*
C9	0.35464 (18)	−0.3034 (3)	0.13187 (16)	0.0332 (5)

H9A	0.2948	−0.3523	0.1254	0.040*
C10	0.43580 (18)	−0.4018 (3)	0.16943 (14)	0.0318 (5)
H10A	0.4282	−0.5142	0.1869	0.038*
C11	0.52649 (17)	−0.3358 (3)	0.18106 (14)	0.0290 (5)
H11A	0.5801	−0.4003	0.2064	0.035*
C12	0.53314 (15)	−0.1673 (3)	0.15278 (13)	0.0233 (4)
C13	0.20664 (14)	0.2894 (3)	0.16465 (13)	0.0202 (4)
C14	0.10780 (14)	0.2457 (3)	0.11280 (12)	0.0202 (4)
C15	0.08006 (15)	0.2934 (3)	0.02933 (13)	0.0214 (4)
H15A	0.1229	0.3486	0.0051	0.026*
C16	−0.01229 (15)	0.2581 (3)	−0.01784 (12)	0.0226 (4)
C17	−0.04769 (16)	0.3129 (3)	−0.10631 (13)	0.0263 (5)
C18	−0.07455 (15)	0.1672 (3)	0.01709 (13)	0.0239 (4)
H18A	−0.1354	0.1415	−0.0150	0.029*
C19	−0.04660 (14)	0.1142 (3)	0.09960 (13)	0.0216 (4)
C20	−0.11372 (14)	0.0066 (3)	0.13342 (13)	0.0238 (4)
C21	0.04383 (14)	0.1585 (3)	0.14761 (12)	0.0207 (4)
H21A	0.0616	0.1293	0.2037	0.025*
N1	0.66800 (12)	0.5074 (2)	0.05512 (11)	0.0248 (4)
N2	0.48148 (12)	0.0938 (2)	0.09546 (11)	0.0238 (4)
N3	0.60976 (12)	−0.0606 (2)	0.15547 (11)	0.0249 (4)
H3B	0.6686	−0.0873	0.1751	0.030*
O1	0.21436 (10)	0.3257 (2)	0.24162 (9)	0.0267 (3)
O2	0.27345 (12)	0.2860 (3)	0.13320 (11)	0.0373 (4)
O3	0.01671 (12)	0.3937 (3)	−0.13642 (10)	0.0334 (4)
H3C	−0.012 (2)	0.424 (5)	−0.1850 (13)	0.059 (10)*
O4	−0.12813 (13)	0.2879 (3)	−0.14653 (11)	0.0483 (5)
O5	−0.19001 (11)	−0.0415 (3)	0.08971 (10)	0.0354 (4)
O6	−0.08317 (11)	−0.0325 (2)	0.21275 (9)	0.0290 (4)
H6B	−0.127 (2)	−0.084 (5)	0.228 (2)	0.072 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.02870 (11)	0.03189 (13)	0.04170 (13)	0.00498 (7)	0.00598 (8)	0.01359 (8)
C1	0.0279 (11)	0.0267 (12)	0.0363 (12)	−0.0075 (9)	0.0050 (9)	0.0005 (10)
C2	0.0318 (12)	0.0334 (14)	0.0365 (13)	−0.0084 (10)	−0.0069 (10)	0.0042 (11)
C3	0.0276 (11)	0.0291 (12)	0.0276 (11)	−0.0021 (9)	−0.0005 (9)	0.0054 (9)
C4	0.0199 (9)	0.0218 (10)	0.0231 (10)	−0.0003 (8)	0.0048 (8)	−0.0011 (8)
C5	0.0198 (9)	0.0272 (11)	0.0247 (10)	0.0001 (8)	0.0037 (8)	0.0025 (9)
C6	0.0211 (9)	0.0228 (11)	0.0215 (9)	0.0001 (8)	0.0042 (7)	0.0002 (8)
C7	0.0227 (10)	0.0238 (11)	0.0245 (10)	−0.0006 (8)	0.0064 (8)	0.0007 (8)
C8	0.0210 (10)	0.0330 (13)	0.0383 (12)	−0.0016 (9)	0.0072 (9)	0.0000 (10)
C9	0.0286 (11)	0.0353 (14)	0.0384 (13)	−0.0099 (10)	0.0133 (10)	−0.0032 (10)
C10	0.0436 (13)	0.0250 (12)	0.0294 (11)	−0.0070 (10)	0.0142 (10)	0.0012 (9)
C11	0.0334 (12)	0.0240 (11)	0.0277 (11)	0.0015 (9)	0.0049 (9)	0.0034 (9)
C12	0.0218 (10)	0.0232 (11)	0.0237 (10)	−0.0004 (8)	0.0035 (8)	−0.0009 (8)
C13	0.0178 (9)	0.0196 (10)	0.0231 (9)	−0.0016 (8)	0.0050 (7)	0.0000 (8)

C14	0.0175 (9)	0.0228 (10)	0.0201 (9)	−0.0001 (8)	0.0045 (7)	−0.0037 (8)
C15	0.0227 (10)	0.0216 (11)	0.0211 (9)	−0.0001 (8)	0.0078 (8)	−0.0008 (8)
C16	0.0243 (10)	0.0241 (11)	0.0184 (9)	0.0008 (8)	0.0036 (8)	−0.0015 (8)
C17	0.0283 (11)	0.0287 (12)	0.0206 (10)	0.0036 (9)	0.0039 (8)	−0.0007 (9)
C18	0.0197 (9)	0.0264 (11)	0.0232 (10)	−0.0002 (8)	0.0014 (8)	−0.0038 (8)
C19	0.0197 (9)	0.0210 (10)	0.0236 (10)	−0.0026 (8)	0.0051 (7)	−0.0025 (8)
C20	0.0205 (9)	0.0246 (11)	0.0265 (10)	0.0003 (8)	0.0064 (8)	−0.0029 (9)
C21	0.0208 (9)	0.0221 (10)	0.0184 (9)	0.0005 (8)	0.0037 (7)	−0.0001 (8)
N1	0.0224 (9)	0.0256 (10)	0.0268 (9)	0.0004 (7)	0.0069 (7)	0.0036 (7)
N2	0.0198 (8)	0.0240 (10)	0.0264 (9)	0.0009 (7)	0.0040 (7)	0.0024 (7)
N3	0.0183 (8)	0.0231 (9)	0.0310 (9)	0.0012 (7)	0.0023 (7)	0.0034 (8)
O1	0.0187 (7)	0.0379 (9)	0.0216 (7)	−0.0007 (6)	0.0017 (6)	−0.0071 (6)
O2	0.0223 (8)	0.0587 (13)	0.0342 (9)	−0.0062 (8)	0.0135 (7)	−0.0057 (8)
O3	0.0291 (8)	0.0483 (11)	0.0225 (8)	0.0030 (8)	0.0061 (6)	0.0082 (7)
O4	0.0313 (9)	0.0744 (15)	0.0297 (9)	−0.0118 (9)	−0.0093 (7)	0.0133 (9)
O5	0.0254 (8)	0.0451 (11)	0.0321 (8)	−0.0132 (7)	0.0010 (6)	0.0028 (8)
O6	0.0237 (8)	0.0391 (10)	0.0239 (7)	−0.0098 (7)	0.0057 (6)	0.0003 (7)

Geometric parameters (Å, °)

Ag1—N2	2.1508 (18)	C11—H11A	0.9300
Ag1—N1^{i}	2.1729 (18)	C12—N3	1.378 (3)
Ag1—O2	2.5320 (17)	C13—O2	1.220 (3)
C1—N1	1.345 (3)	C13—O1	1.289 (3)
C1—C2	1.379 (3)	C13—C14	1.513 (3)
C1—H1A	0.9300	C14—C15	1.393 (3)
C2—C3	1.375 (3)	C14—C21	1.392 (3)
C2—H2A	0.9300	C15—C16	1.398 (3)
C3—C4	1.399 (3)	C15—H15A	0.9300
C3—H3A	0.9300	C16—C18	1.389 (3)
C4—C5	1.389 (3)	C16—C17	1.491 (3)
C4—C6	1.467 (3)	C17—O4	1.205 (3)
C5—N1	1.340 (3)	C17—O3	1.329 (3)
C5—H5A	0.9300	C18—C19	1.390 (3)
C6—N2	1.331 (3)	C18—H18A	0.9300
C6—N3	1.352 (3)	C19—C21	1.395 (3)
C7—N2	1.389 (3)	C19—C20	1.500 (3)
C7—C12	1.398 (3)	C20—O5	1.218 (3)
C7—C8	1.400 (3)	C20—O6	1.316 (3)
C8—C9	1.377 (4)	C21—H21A	0.9300
C8—H8A	0.9300	N3—O1^{ii}	2.828 (2)
C9—C10	1.408 (4)	N3—H3B	0.8600
C9—H9A	0.9300	O3—O6^{iii}	2.772 (2)
C10—C11	1.382 (3)	O3—H3C	0.841 (18)
C10—H10A	0.9300	O6—O1^{iv}	2.487 (2)
C11—C12	1.395 (3)	O6—H6B	0.841 (18)
N2—Ag1—N1^{i}	152.96 (7)	O1—C13—C14	116.17 (17)
N2—Ag1—O2	99.17 (7)	C15—C14—C21	119.42 (18)

N1^{i}—Ag1—O2	107.84 (7)	C15—C14—C13	119.86 (18)
N1—C1—C2	122.1 (2)	C21—C14—C13	120.72 (18)
N1—C1—H1A	119.0	C14—C15—C16	119.83 (19)
C2—C1—H1A	119.0	C14—C15—H15A	120.1
C1—C2—C3	119.9 (2)	C16—C15—H15A	120.1
C1—C2—H2A	120.0	C18—C16—C15	120.08 (19)
C3—C2—H2A	120.0	C18—C16—C17	117.48 (19)
C2—C3—C4	118.8 (2)	C15—C16—C17	122.4 (2)
C2—C3—H3A	120.6	O4—C17—O3	123.1 (2)
C4—C3—H3A	120.6	O4—C17—C16	122.8 (2)
C5—C4—C3	117.7 (2)	O3—C17—C16	114.16 (19)
C5—C4—C6	121.90 (19)	C19—C18—C16	120.49 (19)
C3—C4—C6	120.4 (2)	C19—C18—H18A	119.8
N1—C5—C4	123.40 (19)	C16—C18—H18A	119.8
N1—C5—H5A	118.3	C18—C19—C21	119.07 (19)
C4—C5—H5A	118.3	C18—C19—C20	118.82 (18)
N2—C6—N3	111.94 (19)	C21—C19—C20	122.08 (19)
N2—C6—C4	125.60 (19)	O5—C20—O6	123.3 (2)
N3—C6—C4	122.40 (18)	O5—C20—C19	121.8 (2)
N2—C7—C12	109.27 (18)	O6—C20—C19	114.88 (18)
N2—C7—C8	129.9 (2)	C14—C21—C19	120.96 (19)
C12—C7—C8	120.8 (2)	C14—C21—H21A	119.5
C9—C8—C7	117.1 (2)	C19—C21—H21A	119.5
C9—C8—H8A	121.5	C5—N1—C1	118.03 (19)
C7—C8—H8A	121.5	C5—N1—Ag1^{i}	119.99 (14)
C8—C9—C10	121.8 (2)	C1—N1—Ag1^{i}	121.82 (15)
C8—C9—H9A	119.1	C6—N2—C7	105.49 (18)
C10—C9—H9A	119.1	C6—N2—Ag1	131.45 (15)
C11—C10—C9	121.7 (2)	C7—N2—Ag1	123.03 (14)
C11—C10—H10A	119.1	C6—N3—C12	107.83 (17)
C9—C10—H10A	119.1	C6—N3—O1^{ii}	134.45 (14)
C10—C11—C12	116.3 (2)	C12—N3—O1^{ii}	115.70 (14)
C10—C11—H11A	121.8	C6—N3—H3B	126.1
C12—C11—H11A	121.8	C12—N3—H3B	126.1
N3—C12—C11	132.2 (2)	C13—O2—Ag1	166.85 (16)
N3—C12—C7	105.44 (19)	C17—O3—O6^{iii}	105.52 (13)
C11—C12—C7	122.3 (2)	C17—O3—H3C	106 (2)
O2—C13—O1	123.97 (19)	C20—O6—O1^{iv}	108.75 (13)
O2—C13—C14	119.85 (19)	C20—O6—H6B	109 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) *x*, −*y*+1/2, *z*−1/2; (iv) −*x*, *y*−1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3B···O1^{ii}	0.86	2.01	2.828 (2)	158
O3—H3C···O6^{iii}	0.841 (18)	1.941 (19)	2.772 (2)	169 (4)
O6—H6B···O1^{iv}	0.841 (18)	1.647 (19)	2.487 (2)	176 (4)

Symmetry codes: (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) *x*, −*y*+1/2, *z*−1/2; (iv) −*x*, *y*−1/2, −*z*+1/2.

Fig. 1

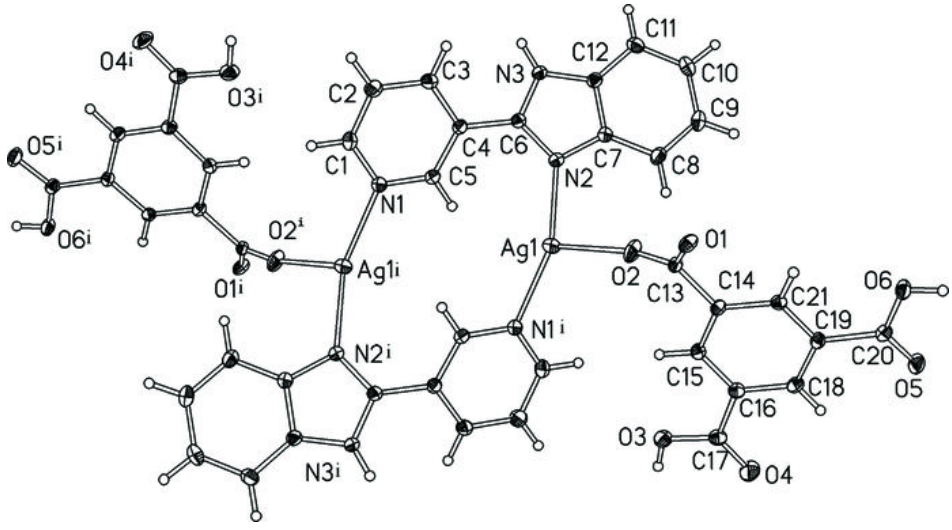

Fig. 2

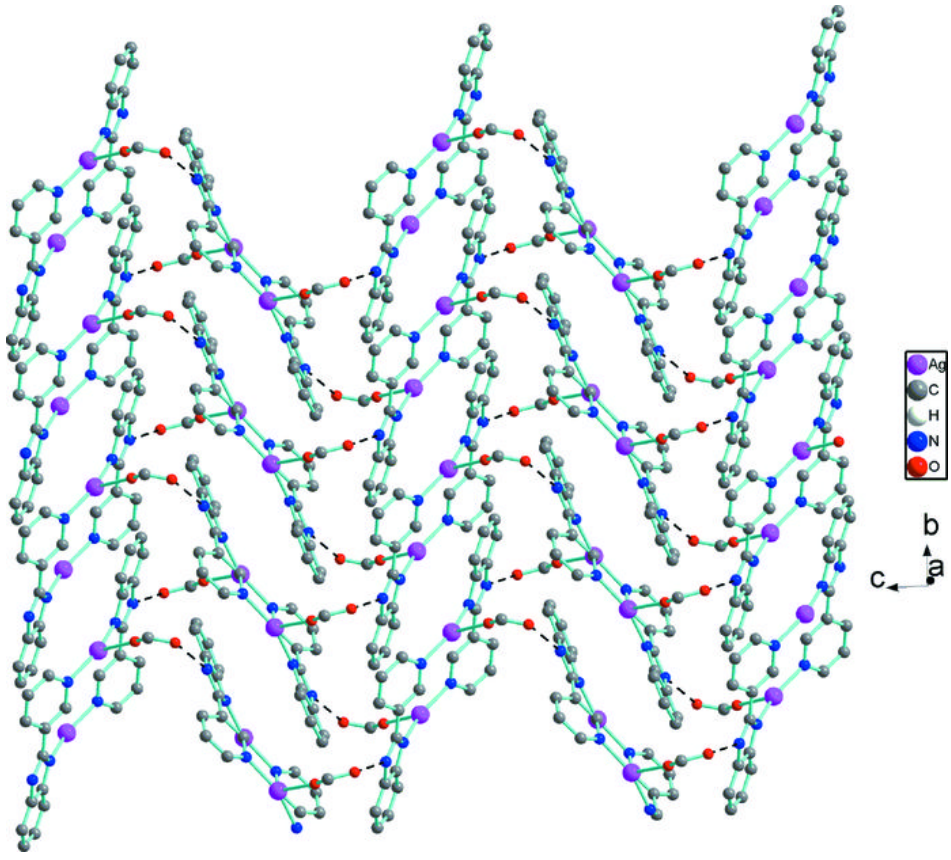

Fig. 3