

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

# Bis[ $\mu$ -1,2-bis(1*H*-imidazol-1-ylmethyl)-benzene- $\kappa^2$ N<sup>3</sup>:N<sup>3'</sup>]disilver(I) bis(4-carboxynaphthalene-1-carboxylate) tetrahydrate

Yan Yang<sup>a\*</sup> and Guohui Yuan<sup>b</sup>

<sup>a</sup>Department of Chemistry, Tonghua Normal University, Tonghua 134001, People's Republic of China, and <sup>b</sup>School of Chemical Engineering and Technology, Harbin Institute of Technology, Harbin 150001, People's Republic of China  
Correspondence e-mail: yangyantonghua@yahoo.com.cn

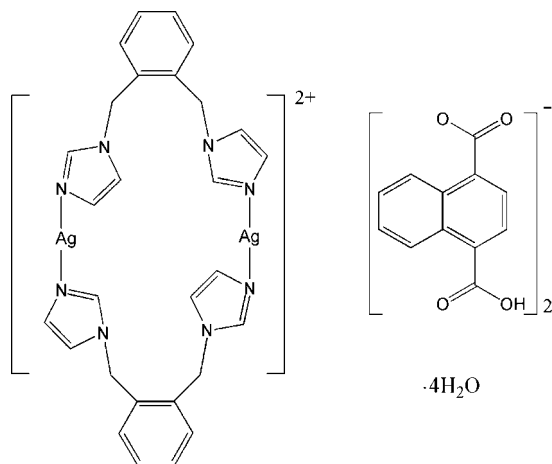
Received 18 April 2011; accepted 17 May 2011

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.029;  $wR$  factor = 0.069; data-to-parameter ratio = 14.2.

In the title compound,  $[\text{Ag}_2(\text{C}_{14}\text{H}_{14}\text{N}_4)_2](\text{C}_{12}\text{H}_7\text{O}_4)_2 \cdot 4\text{H}_2\text{O}$ , the dinuclear dication has crystallographically imposed inversion symmetry. Each  $\text{Ag}^{\text{I}}$  ion is bicoordinated in a slightly distorted linear coordination geometry by the N atoms of two ligands, resulting in the formation of a 22-membered metallamacrocycle. In the dication,  $\pi$ - $\pi$  interactions are observed between the imidazole rings, with centroid-centroid distances of 3.528 (3) Å and dihedral angles of 9.92 (9)°. The crystal structure is stabilized by intermolecular O-H...O hydrogen bonds and  $\pi$ - $\pi$  interactions involving the benzene rings of adjacent dications, with centroid-centroid distances of 3.651 (2) Å.

## Related literature

For the synthesis and structures of related compounds, see: Tan *et al.* (2004); Liu *et al.* (2007); Liu, Ma *et al.* (2008); Liu, Chi & Wang (2008); Sun *et al.* (2009).



## Experimental

### Crystal data

$[\text{Ag}_2(\text{C}_{14}\text{H}_{14}\text{N}_4)_2](\text{C}_{12}\text{H}_7\text{O}_4)_2 \cdot 4\text{H}_2\text{O}$   
 $M_r = 1194.74$   
 Triclinic,  $P\bar{1}$   
 $a = 9.6644$  (5) Å  
 $b = 11.3769$  (12) Å  
 $c = 11.8255$  (5) Å  
 $\alpha = 109.376$  (8)°  
 $\beta = 95.783$  (3)°  
 $\gamma = 94.442$  (4)°  
 $V = 1211.79$  (15) Å<sup>3</sup>  
 $Z = 1$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.88$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.15 \times 0.12 \times 0.11$  mm

### Data collection

Bruker APEX diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 1999)  
 $T_{\text{min}} = 0.35$ ,  $T_{\text{max}} = 0.59$   
 8572 measured reflections  
 4904 independent reflections  
 3384 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.023$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.069$   
 $S = 0.89$   
 4904 reflections  
 346 parameters  
 6 restraints  
 H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.43$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------|----------|-------------|-------------|---------------|
| O2-H2A...O3 <sup>i</sup>     | 0.82     | 1.69        | 2.496 (2)   | 166           |
| O1W-HW11...O4                | 0.87 (2) | 1.96 (2)    | 2.814 (3)   | 166 (3)       |
| O1W-HW12...O2W <sup>ii</sup> | 0.83 (2) | 2.12 (2)    | 2.902 (3)   | 158 (3)       |
| O2W-HW21...O1                | 0.84 (2) | 1.99 (2)    | 2.810 (3)   | 164 (4)       |
| O2W-HW22...O3 <sup>i</sup>   | 0.88 (2) | 2.13 (3)    | 2.841 (3)   | 138 (3)       |

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

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

The authors thank Tonghua Normal University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2589).

## References

- Bruker (1997). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Bruker (1999). SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Liu, H.-Y., Chi, Y.-C. & Wang, G.-H. (2008). *Acta Cryst.* E64, m1071.  
 Liu, Y.-Y., Ma, J.-C., Zhang, L.-P. & Ma, J.-F. (2008). *J. Coord. Chem.* 61, 3583-3593.  
 Liu, H.-Y., Sun, H.-M. & Ma, J.-F. (2007). *Acta Cryst.* E63, m3109.  
 Sheldrick, G. M. (2008). *Acta Cryst.* A64, 112-122.  
 Sun, H.-M., Chi, Y.-C. & Liu, H.-Y. (2009). *Acta Cryst.* E65, m1042-m1043.  
 Tan, H.-Y., Zhang, H.-X., Ou, H.-D. & Kang, B.-S. (2004). *Inorg. Chim. Acta*, 357, 869-874.

## supporting information

*Acta Cryst.* (2011). E67, m802 [doi:10.1107/S1600536811018691]

## Bis[ $\mu$ -1,2-bis(1*H*-imidazol-1-ylmethyl)benzene- $\kappa^2$ N<sup>3</sup>:N<sup>3'</sup>]]disilver(I) bis(4-carboxynaphthalene-1-carboxylate) tetrahydrate

Yan Yang and Guohui Yuan

### S1. Comment

The design and synthesis of silver(I) complexes have attracted intense interests of chemists (Liu, Chi & Wang, 2008; Tan *et al.*, 2004) because of the versatility of their coordination geometry (Sun *et al.*, 2009). So far, some complexes, modified by secondary nitrogen-based ligands, have been reported (Liu *et al.*, 2007). In this work, the combination of 1,2-bis(1*H*-imidazol-1-ylmethyl)benzene (1,2-bix) with naphthalene-1,4-dicarboxylic acid (1,4-H<sub>2</sub>ndc) and silver(I) ions resulted in the title compound, whose synthesis and structure are reported herein.

The contents of the asymmetric unit of the title compound is shown in Fig. 1. The complex, which has crystallographically imposed inversion symmetry, shows a binuclear structure, where each of silver(I) atom has a slightly distorted linear geometry and is coordinated by the N atoms from two 1,2-bix ligands. The Ag-N bond distances are within the normal range and are comparable to those observed in related N-containing compounds (Liu, Ma *et al.*, 2008). Notably, the 1,4-H<sub>2</sub>ndc anion does not coordinate to the metal and acts as a counter-anion. In the dication,  $\pi$ - $\pi$  interactions are observed between the imidazole rings (N1/N2/C1-C3 and N3/N4/C12-C14), with centroid-centroid distance of 3.528 (3) Å and dihedral angles of 9.92 (9)°. The crystal structure is stabilized by a three-dimensional network of intermolecular O—H $\cdots$ O hydrogen bonds (Table 1) and  $\pi$ - $\pi$  interactions involving the benzene rings of adjacent dications, with centroid-to-centroid distances  $Cg1\cdots Cg1^i = 3.651$  (2) Å [ $Cg1$  is the centroid of the C5-C10 ring; symmetry code: (i) 1-x, -y, 1-z].

### S2. Experimental

A mixture of AgNO<sub>3</sub>·2H<sub>2</sub>O (0.5 mmol), naphthalene-1,4-dicarboxylic acid (0.5 mmol), 1,2-bis(1*H*-imidazol-1-ylmethyl)benzene (0.5 mmol) in H<sub>2</sub>O (12 ml) was adjusted to pH = 5-6 by addition of aqueous NaOH solution, and heated at 145°C for 2 days. After the mixture was slowly cooled to room temperature, crystals of the title compound suitable for X-ray analysis were obtained (yield 33%).

### S3. Refinement

Water hydrogen atoms were located in difference Fourier maps and refined isotropically, with distance restraints of O—H = 0.85 (1) and H $\cdots$ H = 1.35 (1) Å and with  $U_{iso}(H) = 1.5U_{eq}(O)$ . All other H atoms were positioned geometrically (C—H = 0.93 Å, O—H = 0.82 Å) and refined as riding, with  $U_{iso}(H) = 1.2U_{eq}(C, O)$ .

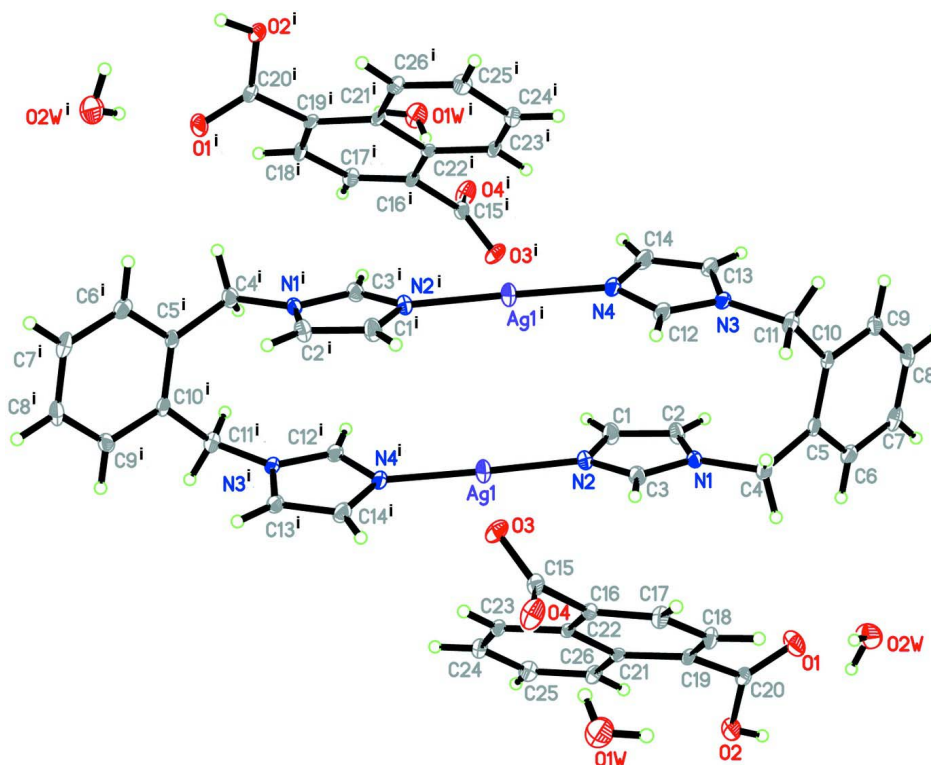


Figure 1

The structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level. Symmetry code: (i) 2-x, -y, -z.

**Bis[ $\mu$ -1,2-bis(1*H*-imidazol-1-ylmethyl)benzene- $\kappa^2N^3:N^3'$ ]disilver(I) bis(4-carboxynaphthalene-1-carboxylate) tetrahydrate**

*Crystal data*

[Ag<sub>2</sub>(C<sub>14</sub>H<sub>14</sub>N<sub>4</sub>)<sub>2</sub>](C<sub>12</sub>H<sub>7</sub>O<sub>4</sub>)<sub>2</sub>·4H<sub>2</sub>O

*M<sub>r</sub>* = 1194.74

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

*a* = 9.6644 (5) Å

*b* = 11.3769 (12) Å

*c* = 11.8255 (5) Å

$\alpha$  = 109.376 (8)°

$\beta$  = 95.783 (3)°

$\gamma$  = 94.442 (4)°

*V* = 1211.79 (15) Å<sup>3</sup>

*Z* = 1

*F*(000) = 608

*D<sub>x</sub>* = 1.637 Mg m<sup>-3</sup>

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

Cell parameters from 4904 reflections

$\theta$  = 1.8–26.4°

$\mu$  = 0.88 mm<sup>-1</sup>

*T* = 293 K

Block, pale yellow

0.15 × 0.12 × 0.11 mm

*Data collection*

Bruker APEX  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 1999)

*T<sub>min</sub>* = 0.35, *T<sub>max</sub>* = 0.59

8572 measured reflections

4904 independent reflections

3384 reflections with *I* > 2 $\sigma$ (*I*)

*R<sub>int</sub>* = 0.023

$\theta_{\max}$  = 26.4°,  $\theta_{\min}$  = 1.8°

*h* = -11→12

*k* = -14→12

*l* = -14→11

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.069$   
 $S = 0.89$   
 4904 reflections  
 346 parameters  
 6 restraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0385P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.30 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.43 \text{ e } \text{Å}^{-3}$

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{Å}^2$ )*

|      | <i>x</i>   | <i>y</i>    | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|------------|-------------|--------------|----------------------------------|
| C1   | 0.6791 (3) | -0.0550 (2) | 0.0091 (2)   | 0.0501 (6)                       |
| H1   | 0.6710     | -0.1140     | -0.0684      | 0.060*                           |
| C2   | 0.6045 (3) | -0.0634 (2) | 0.0974 (2)   | 0.0458 (6)                       |
| H2   | 0.5361     | -0.1279     | 0.0919       | 0.055*                           |
| C3   | 0.7469 (2) | 0.1091 (2)  | 0.1643 (2)   | 0.0424 (6)                       |
| H3   | 0.7937     | 0.1857      | 0.2157       | 0.051*                           |
| C4   | 0.5997 (3) | 0.0755 (2)  | 0.3151 (2)   | 0.0442 (6)                       |
| H4A  | 0.5347     | 0.1374      | 0.3205       | 0.053*                           |
| H4B  | 0.6790     | 0.1137      | 0.3774       | 0.053*                           |
| C5   | 0.5284 (2) | -0.0355 (2) | 0.33815 (18) | 0.0341 (5)                       |
| C6   | 0.3839 (2) | -0.0568 (2) | 0.3188 (2)   | 0.0434 (6)                       |
| H6   | 0.3335     | -0.0034     | 0.2905       | 0.052*                           |
| C7   | 0.3121 (2) | -0.1558 (3) | 0.3405 (2)   | 0.0479 (6)                       |
| H7   | 0.2147     | -0.1685     | 0.3266       | 0.058*                           |
| C8   | 0.3846 (3) | -0.2340 (2) | 0.3823 (2)   | 0.0460 (6)                       |
| H8   | 0.3371     | -0.3008     | 0.3968       | 0.055*                           |
| C9   | 0.5289 (3) | -0.2138 (2) | 0.4032 (2)   | 0.0420 (6)                       |
| H9   | 0.5780     | -0.2673     | 0.4324       | 0.050*                           |
| C10  | 0.6025 (2) | -0.1153 (2) | 0.38161 (18) | 0.0329 (5)                       |
| C11  | 0.7610 (2) | -0.0995 (2) | 0.4061 (2)   | 0.0443 (6)                       |
| H11A | 0.7950     | -0.0110     | 0.4437       | 0.053*                           |
| H11B | 0.7903     | -0.1418     | 0.4618       | 0.053*                           |
| C12  | 0.9213 (2) | -0.0917 (2) | 0.2549 (2)   | 0.0402 (6)                       |
| H12  | 0.9636     | -0.0104     | 0.2967       | 0.048*                           |

|      |              |               |              |              |
|------|--------------|---------------|--------------|--------------|
| C13  | 0.7869 (2)   | -0.2677 (2)   | 0.2088 (2)   | 0.0465 (6)   |
| H13  | 0.7210       | -0.3304       | 0.2117       | 0.056*       |
| C14  | 0.8654 (3)   | -0.2743 (2)   | 0.1202 (2)   | 0.0510 (7)   |
| H14  | 0.8624       | -0.3432       | 0.0500       | 0.061*       |
| C15  | 1.0338 (2)   | 0.4758 (2)    | 0.2815 (2)   | 0.0376 (5)   |
| C16  | 0.87617 (19) | 0.45208 (19)  | 0.27283 (19) | 0.0296 (5)   |
| C17  | 0.8186 (2)   | 0.4650 (2)    | 0.3765 (2)   | 0.0392 (6)   |
| H17  | 0.8761       | 0.4916        | 0.4503       | 0.047*       |
| C18  | 0.6738 (2)   | 0.4386 (2)    | 0.3729 (2)   | 0.0385 (5)   |
| H18  | 0.6374       | 0.4426        | 0.4438       | 0.046*       |
| C19  | 0.58560 (19) | 0.40728 (19)  | 0.26764 (19) | 0.0294 (5)   |
| C20  | 0.4319 (2)   | 0.3805 (2)    | 0.2734 (2)   | 0.0353 (5)   |
| C21  | 0.64007 (19) | 0.39844 (18)  | 0.15716 (18) | 0.0255 (4)   |
| C22  | 0.78814 (19) | 0.41758 (18)  | 0.16013 (18) | 0.0246 (4)   |
| C23  | 0.8428 (2)   | 0.40779 (19)  | 0.05138 (19) | 0.0314 (5)   |
| H23  | 0.9393       | 0.4196        | 0.0527       | 0.038*       |
| C24  | 0.7584 (2)   | 0.3816 (2)    | -0.0548 (2)  | 0.0386 (5)   |
| H24  | 0.7967       | 0.3765        | -0.1251      | 0.046*       |
| C25  | 0.6131 (2)   | 0.3624 (2)    | -0.0577 (2)  | 0.0417 (6)   |
| H25  | 0.5551       | 0.3439        | -0.1306      | 0.050*       |
| C26  | 0.5555 (2)   | 0.3705 (2)    | 0.04427 (19) | 0.0357 (5)   |
| H26  | 0.4587       | 0.3575        | 0.0400       | 0.043*       |
| O1   | 0.39354 (17) | 0.3166 (2)    | 0.3301 (2)   | 0.0755 (7)   |
| O2   | 0.35024 (15) | 0.43133 (18)  | 0.21712 (17) | 0.0592 (5)   |
| H2A  | 0.2689       | 0.4124        | 0.2246       | 0.089*       |
| O1W  | 1.0555 (2)   | 0.7995 (2)    | 0.5309 (2)   | 0.0790 (6)   |
| HW11 | 1.053 (4)    | 0.725 (2)     | 0.477 (3)    | 0.119*       |
| HW12 | 0.991 (3)    | 0.788 (3)     | 0.569 (3)    | 0.119*       |
| O3   | 1.09219 (15) | 0.38750 (17)  | 0.21193 (16) | 0.0525 (4)   |
| O2W  | 0.1287 (2)   | 0.1769 (2)    | 0.2855 (3)   | 0.0908 (7)   |
| HW21 | 0.204 (3)    | 0.224 (3)     | 0.313 (4)    | 0.136*       |
| HW22 | 0.086 (4)    | 0.210 (4)     | 0.237 (3)    | 0.136*       |
| O4   | 1.09571 (17) | 0.57350 (19)  | 0.35306 (17) | 0.0630 (5)   |
| Ag1  | 0.91046 (2)  | 0.11314 (2)   | -0.04497 (2) | 0.05935 (10) |
| N1   | 0.64894 (18) | 0.04051 (16)  | 0.19530 (16) | 0.0346 (4)   |
| N2   | 0.7687 (2)   | 0.05428 (19)  | 0.05184 (18) | 0.0461 (5)   |
| N3   | 0.82247 (17) | -0.15126 (17) | 0.29391 (16) | 0.0350 (4)   |
| N4   | 0.95103 (19) | -0.16342 (18) | 0.14922 (18) | 0.0443 (5)   |

Atomic displacement parameters ( $\text{\AA}^2$ )

|    | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$    |
|----|-------------|-------------|-------------|--------------|-------------|-------------|
| C1 | 0.0624 (17) | 0.0469 (16) | 0.0398 (14) | 0.0010 (14)  | 0.0177 (12) | 0.0115 (12) |
| C2 | 0.0520 (15) | 0.0433 (15) | 0.0415 (14) | -0.0066 (12) | 0.0128 (11) | 0.0145 (12) |
| C3 | 0.0452 (14) | 0.0336 (13) | 0.0498 (16) | -0.0008 (11) | 0.0144 (12) | 0.0151 (12) |
| C4 | 0.0566 (15) | 0.0378 (14) | 0.0404 (14) | 0.0063 (12)  | 0.0215 (12) | 0.0118 (11) |
| C5 | 0.0379 (13) | 0.0377 (13) | 0.0275 (12) | 0.0048 (10)  | 0.0158 (10) | 0.0089 (10) |
| C6 | 0.0396 (13) | 0.0542 (16) | 0.0362 (13) | 0.0137 (12)  | 0.0109 (11) | 0.0118 (12) |

|     |              |              |              |              |              |              |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| C7  | 0.0315 (13)  | 0.0632 (18)  | 0.0379 (14)  | -0.0060 (12) | 0.0124 (11)  | 0.0029 (13)  |
| C8  | 0.0487 (15)  | 0.0441 (15)  | 0.0401 (14)  | -0.0110 (13) | 0.0201 (12)  | 0.0073 (12)  |
| C9  | 0.0525 (15)  | 0.0419 (14)  | 0.0365 (13)  | 0.0076 (12)  | 0.0177 (11)  | 0.0162 (11)  |
| C10 | 0.0337 (12)  | 0.0372 (13)  | 0.0272 (11)  | 0.0032 (10)  | 0.0134 (9)   | 0.0080 (10)  |
| C11 | 0.0362 (13)  | 0.0569 (16)  | 0.0398 (14)  | 0.0055 (12)  | 0.0122 (11)  | 0.0143 (12)  |
| C12 | 0.0309 (12)  | 0.0368 (13)  | 0.0545 (16)  | 0.0030 (10)  | 0.0133 (11)  | 0.0156 (12)  |
| C13 | 0.0425 (14)  | 0.0290 (13)  | 0.0708 (18)  | 0.0033 (11)  | 0.0266 (13)  | 0.0157 (13)  |
| C14 | 0.0514 (15)  | 0.0351 (14)  | 0.0646 (17)  | 0.0075 (12)  | 0.0272 (13)  | 0.0084 (13)  |
| C15 | 0.0227 (11)  | 0.0521 (15)  | 0.0397 (14)  | -0.0015 (11) | 0.0024 (10)  | 0.0198 (12)  |
| C16 | 0.0181 (10)  | 0.0340 (12)  | 0.0381 (13)  | 0.0033 (9)   | 0.0047 (9)   | 0.0138 (10)  |
| C17 | 0.0228 (11)  | 0.0583 (16)  | 0.0344 (13)  | 0.0021 (11)  | -0.0020 (9)  | 0.0155 (12)  |
| C18 | 0.0269 (11)  | 0.0567 (15)  | 0.0342 (13)  | 0.0049 (11)  | 0.0103 (10)  | 0.0166 (11)  |
| C19 | 0.0186 (10)  | 0.0339 (12)  | 0.0369 (13)  | 0.0056 (9)   | 0.0070 (9)   | 0.0122 (10)  |
| C20 | 0.0224 (11)  | 0.0426 (14)  | 0.0408 (13)  | 0.0032 (10)  | 0.0089 (10)  | 0.0131 (11)  |
| C21 | 0.0194 (10)  | 0.0240 (11)  | 0.0330 (12)  | 0.0043 (8)   | 0.0046 (9)   | 0.0090 (9)   |
| C22 | 0.0191 (10)  | 0.0214 (10)  | 0.0321 (12)  | 0.0019 (8)   | 0.0042 (8)   | 0.0076 (9)   |
| C23 | 0.0212 (10)  | 0.0320 (12)  | 0.0413 (13)  | 0.0025 (9)   | 0.0105 (10)  | 0.0114 (10)  |
| C24 | 0.0377 (13)  | 0.0426 (14)  | 0.0344 (13)  | 0.0022 (11)  | 0.0095 (10)  | 0.0109 (11)  |
| C25 | 0.0385 (13)  | 0.0503 (15)  | 0.0303 (13)  | 0.0017 (11)  | -0.0035 (10) | 0.0092 (11)  |
| C26 | 0.0216 (11)  | 0.0423 (14)  | 0.0393 (13)  | 0.0015 (10)  | 0.0028 (10)  | 0.0095 (11)  |
| O1  | 0.0282 (9)   | 0.1157 (17)  | 0.1216 (18)  | 0.0090 (10)  | 0.0207 (10)  | 0.0889 (15)  |
| O2  | 0.0200 (8)   | 0.0928 (14)  | 0.0906 (14)  | 0.0163 (9)   | 0.0166 (9)   | 0.0605 (12)  |
| O1W | 0.0747 (15)  | 0.0615 (14)  | 0.0900 (17)  | -0.0045 (12) | 0.0228 (12)  | 0.0110 (12)  |
| O3  | 0.0211 (8)   | 0.0636 (12)  | 0.0697 (12)  | 0.0109 (8)   | 0.0091 (8)   | 0.0167 (10)  |
| O2W | 0.0619 (14)  | 0.0779 (17)  | 0.146 (2)    | -0.0041 (12) | 0.0282 (14)  | 0.0553 (16)  |
| O4  | 0.0303 (9)   | 0.0721 (13)  | 0.0657 (12)  | -0.0156 (9)  | 0.0044 (9)   | 0.0014 (11)  |
| Ag1 | 0.05254 (14) | 0.07269 (17) | 0.07007 (17) | 0.00896 (11) | 0.03247 (11) | 0.03988 (13) |
| N1  | 0.0398 (11)  | 0.0306 (10)  | 0.0364 (11)  | 0.0028 (9)   | 0.0122 (8)   | 0.0139 (9)   |
| N2  | 0.0498 (12)  | 0.0460 (13)  | 0.0540 (14)  | 0.0089 (10)  | 0.0240 (10)  | 0.0268 (11)  |
| N3  | 0.0284 (10)  | 0.0357 (11)  | 0.0442 (11)  | 0.0057 (8)   | 0.0121 (8)   | 0.0155 (9)   |
| N4  | 0.0412 (11)  | 0.0414 (12)  | 0.0590 (14)  | 0.0097 (10)  | 0.0258 (10)  | 0.0221 (10)  |

*Geometric parameters (Å, °)*

|        |           |         |           |
|--------|-----------|---------|-----------|
| C1—C2  | 1.351 (3) | C14—H14 | 0.9300    |
| C1—N2  | 1.371 (3) | C15—O4  | 1.217 (3) |
| C1—H1  | 0.9300    | C15—O3  | 1.279 (3) |
| C2—N1  | 1.355 (3) | C15—C16 | 1.513 (3) |
| C2—H2  | 0.9300    | C16—C17 | 1.365 (3) |
| C3—N2  | 1.315 (3) | C16—C22 | 1.424 (3) |
| C3—N1  | 1.336 (3) | C17—C18 | 1.402 (3) |
| C3—H3  | 0.9300    | C17—H17 | 0.9300    |
| C4—N1  | 1.477 (3) | C18—C19 | 1.360 (3) |
| C4—C5  | 1.508 (3) | C18—H18 | 0.9300    |
| C4—H4A | 0.9700    | C19—C21 | 1.433 (3) |
| C4—H4B | 0.9700    | C19—C20 | 1.506 (3) |
| C5—C6  | 1.381 (3) | C20—O1  | 1.203 (3) |
| C5—C10 | 1.393 (3) | C20—O2  | 1.273 (3) |

|            |             |                     |             |
|------------|-------------|---------------------|-------------|
| C6—C7      | 1.387 (3)   | C21—C26             | 1.417 (3)   |
| C6—H6      | 0.9300      | C21—C22             | 1.426 (3)   |
| C7—C8      | 1.359 (4)   | C22—C23             | 1.413 (3)   |
| C7—H7      | 0.9300      | C23—C24             | 1.356 (3)   |
| C8—C9      | 1.379 (3)   | C23—H23             | 0.9300      |
| C8—H8      | 0.9300      | C24—C25             | 1.400 (3)   |
| C9—C10     | 1.389 (3)   | C24—H24             | 0.9300      |
| C9—H9      | 0.9300      | C25—C26             | 1.357 (3)   |
| C10—C11    | 1.515 (3)   | C25—H25             | 0.9300      |
| C11—N3     | 1.468 (3)   | C26—H26             | 0.9300      |
| C11—H11A   | 0.9700      | O2—H2A              | 0.8200      |
| C11—H11B   | 0.9700      | O1W—HW11            | 0.874 (17)  |
| C12—N4     | 1.320 (3)   | O1W—HW12            | 0.833 (17)  |
| C12—N3     | 1.334 (3)   | O2W—HW21            | 0.838 (18)  |
| C12—H12    | 0.9300      | O2W—HW22            | 0.882 (18)  |
| C13—C14    | 1.340 (3)   | Ag1—N2              | 2.0783 (17) |
| C13—N3     | 1.364 (3)   | Ag1—N4 <sup>i</sup> | 2.0787 (17) |
| C13—H13    | 0.9300      | N4—Ag1 <sup>i</sup> | 2.0787 (17) |
| C14—N4     | 1.373 (3)   |                     |             |
|            |             |                     |             |
| C2—C1—N2   | 109.2 (2)   | O3—C15—C16          | 115.7 (2)   |
| C2—C1—H1   | 125.4       | C17—C16—C22         | 119.93 (18) |
| N2—C1—H1   | 125.4       | C17—C16—C15         | 118.49 (18) |
| C1—C2—N1   | 106.6 (2)   | C22—C16—C15         | 121.58 (17) |
| C1—C2—H2   | 126.7       | C16—C17—C18         | 120.8 (2)   |
| N1—C2—H2   | 126.7       | C16—C17—H17         | 119.6       |
| N2—C3—N1   | 111.2 (2)   | C18—C17—H17         | 119.6       |
| N2—C3—H3   | 124.4       | C19—C18—C17         | 121.12 (19) |
| N1—C3—H3   | 124.4       | C19—C18—H18         | 119.4       |
| N1—C4—C5   | 112.57 (18) | C17—C18—H18         | 119.4       |
| N1—C4—H4A  | 109.1       | C18—C19—C21         | 120.19 (17) |
| C5—C4—H4A  | 109.1       | C18—C19—C20         | 117.02 (18) |
| N1—C4—H4B  | 109.1       | C21—C19—C20         | 122.78 (18) |
| C5—C4—H4B  | 109.1       | O1—C20—O2           | 124.30 (19) |
| H4A—C4—H4B | 107.8       | O1—C20—C19          | 120.2 (2)   |
| C6—C5—C10  | 118.62 (19) | O2—C20—C19          | 115.45 (18) |
| C6—C5—C4   | 118.8 (2)   | C26—C21—C22         | 117.74 (17) |
| C10—C5—C4  | 122.5 (2)   | C26—C21—C19         | 123.92 (17) |
| C5—C6—C7   | 121.6 (2)   | C22—C21—C19         | 118.34 (17) |
| C5—C6—H6   | 119.2       | C23—C22—C16         | 121.85 (17) |
| C7—C6—H6   | 119.2       | C23—C22—C21         | 118.69 (18) |
| C8—C7—C6   | 119.7 (2)   | C16—C22—C21         | 119.38 (17) |
| C8—C7—H7   | 120.2       | C24—C23—C22         | 121.85 (19) |
| C6—C7—H7   | 120.2       | C24—C23—H23         | 119.1       |
| C7—C8—C9   | 119.7 (2)   | C22—C23—H23         | 119.1       |
| C7—C8—H8   | 120.2       | C23—C24—C25         | 119.4 (2)   |
| C9—C8—H8   | 120.2       | C23—C24—H24         | 120.3       |
| C8—C9—C10  | 121.4 (2)   | C25—C24—H24         | 120.3       |

|               |             |                         |             |
|---------------|-------------|-------------------------|-------------|
| C8—C9—H9      | 119.3       | C26—C25—C24             | 121.0 (2)   |
| C10—C9—H9     | 119.3       | C26—C25—H25             | 119.5       |
| C9—C10—C5     | 119.0 (2)   | C24—C25—H25             | 119.5       |
| C9—C10—C11    | 118.4 (2)   | C25—C26—C21             | 121.34 (19) |
| C5—C10—C11    | 122.57 (19) | C25—C26—H26             | 119.3       |
| N3—C11—C10    | 111.23 (18) | C21—C26—H26             | 119.3       |
| N3—C11—H11A   | 109.4       | C20—O2—H2A              | 109.5       |
| C10—C11—H11A  | 109.4       | HW11—O1W—HW12           | 101 (2)     |
| N3—C11—H11B   | 109.4       | HW21—O2W—HW22           | 103 (2)     |
| C10—C11—H11B  | 109.4       | N2—Ag1—N4 <sup>i</sup>  | 177.04 (8)  |
| H11A—C11—H11B | 108.0       | C3—N1—C2                | 107.41 (18) |
| N4—C12—N3     | 111.1 (2)   | C3—N1—C4                | 124.93 (19) |
| N4—C12—H12    | 124.5       | C2—N1—C4                | 127.65 (18) |
| N3—C12—H12    | 124.5       | C3—N2—C1                | 105.65 (18) |
| C14—C13—N3    | 106.6 (2)   | C3—N2—Ag1               | 128.88 (16) |
| C14—C13—H13   | 126.7       | C1—N2—Ag1               | 125.46 (16) |
| N3—C13—H13    | 126.7       | C12—N3—C13              | 107.32 (19) |
| C13—C14—N4    | 109.5 (2)   | C12—N3—C11              | 126.2 (2)   |
| C13—C14—H14   | 125.3       | C13—N3—C11              | 126.49 (18) |
| N4—C14—H14    | 125.3       | C12—N4—C14              | 105.53 (18) |
| O4—C15—O3     | 124.9 (2)   | C12—N4—Ag1 <sup>i</sup> | 126.65 (15) |
| O4—C15—C16    | 119.4 (2)   | C14—N4—Ag1 <sup>i</sup> | 127.71 (16) |

Symmetry code: (i)  $-x+2, -y, -z$ .

*Hydrogen-bond geometry (Å, °)*

| <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> |
|--------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O2—H2A $\cdots$ O3 <sup>ii</sup>     | 0.82        | 1.69                | 2.496 (2)                  | 166                           |
| O1W—HW11 $\cdots$ O4                 | 0.87 (2)    | 1.96 (2)            | 2.814 (3)                  | 166 (3)                       |
| O1W—HW12 $\cdots$ O2W <sup>iii</sup> | 0.83 (2)    | 2.12 (2)            | 2.902 (3)                  | 158 (3)                       |
| O2W—HW21 $\cdots$ O1                 | 0.84 (2)    | 1.99 (2)            | 2.810 (3)                  | 164 (4)                       |
| O2W—HW22 $\cdots$ O3 <sup>ii</sup>   | 0.88 (2)    | 2.13 (3)            | 2.841 (3)                  | 138 (3)                       |

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