



Crystal structure of bis[1-(2-hydroxyethyl)-2-methyl-5-nitro-1*H*-imidazole- κ N³]silver(I) tetrafluoridoborate methanol monosolvate

Joshua H. Palmer^a and Rita K. Upmacis^{b*}

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^aDepartment of Chemistry, Columbia University, New York, NY 10027, USA, and ^bHaskins Laboratories, Dept. of Chemistry, Pace University, New York, NY 10038, USA. *Correspondence e-mail: rupmacis@pace.edu

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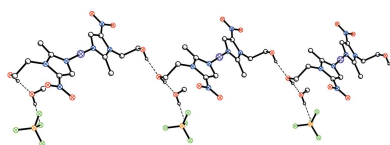
1-(2-Hydroxyethyl)-2-methyl-5-nitro-1*H*-imidazole (metronidazole, MET) is a medication that is used to treat infections by a variety of anaerobic organisms, but there are relatively few reports of the structures of metal compounds that exhibit coordination of metronidazole. We have demonstrated that MET reacts with AgBF₄ to give [Ag(MET)₂]BF₄·CH₃OH, in which the Ag^I cation is coordinated by two MET ligands with a *trans* arrangement. The structure of [Ag(MET)₂]BF₄ exhibits some interesting differences from its nitrate counterpart, [Ag(MET)₂]NO₃ [Fun *et al.* (2008). *Acta Cryst. E* **64**, m668]. For instance, although the two MET ligands of both [Ag(MET)₂]BF₄ and [Ag(MET)₂]NO₃ are almost coplanar, the former compound has an *anti*-like geometry with a molecular inversion center, but the latter has a *syn*-like arrangement. In the crystal, the BF₄[−] anion is linked by an O—H···F hydrogen bond to the methanol solvent molecule, which is, in turn, linked to the cation by an O—H···O hydrogen bond; the components of the structure are linked by O—H···O hydrogen bonds, forming chains along [001]. One of the MET ligands and the BF₄[−] anion are disordered over two sets of sites with ratios of refined occupancies 0.501 (17):0.499 (17) and 0.539 (19):0.461 (19), respectively.

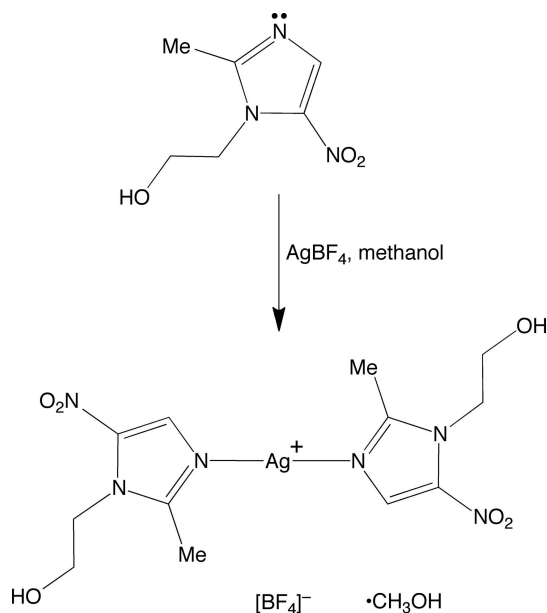
1. Chemical context

1-(2-Hydroxyethyl)-2-methyl-5-nitro-1*H*-imidazole, also known as metronidazole (MET) or Flagyl, is a medication used particularly for treatment of parasitic infections, such as trichomoniasis, amoebiasis and giardiasis, but is also effective against anaerobic bacteria (Freeman *et al.*, 1997; Miljkovic *et al.*, 2014; Soares *et al.*, 2012; Samuelson, 1999; Lofmark *et al.*, 2010). There are relatively few reports of the structures of metal compounds that exhibit coordination of MET. For example, with respect to silver, only the nitrate compound, [Ag(MET)₂]NO₃, has been structurally characterized by X-ray diffraction (Fun *et al.*, 2008). Herein, we describe the structure of the tetrafluoridoborate derivative, [Ag(MET)₂]BF₄, which is obtained by addition of MET to AgBF₄ in methanol (see Scheme).

2. Structural commentary

Crystals of composition [Ag(MET)₂]BF₄·MeOH were obtained from a solution in methanol. The asymmetric unit consists of a silver cation, [Ag(MET)₂]⁺, a tetrafluoridoborate anion, BF₄[−], and a solvent methanol molecule. The silver atom of [Ag(MET)₂]⁺ is coordinated by two MET ligands in a *trans* manner by their N³ nitrogen atoms, as illustrated in Fig. 1.





One of the MET ligands exhibits disorder resulting from rotation about the Ag–N bond [the dihedral angle between the planes of the disordered 5-membered rings is 11.0 (9)°]. The Ag–N bond lengths [Ag–N11 = 2.082 (15), Ag–N11A = 2.163 (16) and Ag–N21 = 2.1193 (15) Å] are comparable to those values in the nitrate derivative, [2.1489 (11) and 2.1475 (11) Å; Fun *et al.*, 2008]. There are, however, some interesting differences between the two compounds.

First, while the two MET ligands of both [Ag(MET)₂]BF₄ and [Ag(MET)₂]NO₃ are almost coplanar, the former compound has an *anti*-like geometry, and the latter has a *syn*-like arrangement. Thus, the C13–N11···N21–C23 torsion angle for [Ag(MET)₂]BF₄ is 160.8 (9)° [148.6 (11)° for the minor component of disorder], while the value for [Ag(MET)₂]NO₃ is 24.10° (Fun *et al.*, 2008). These differences are illustrated in Fig. 2, which shows that the [Ag(MET)₂]⁺ unit of [Ag(MET)₂]BF₄ has an approximate inversion center at the Ag^I ion, whereas [Ag(MET)₂]NO₃ does not.

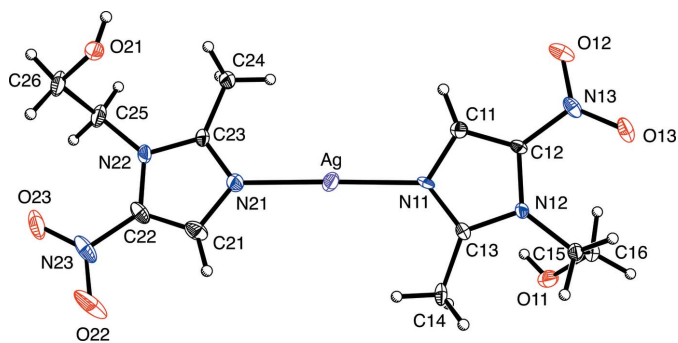


Figure 1
The molecular structure of the cation of the title compound, with displacement ellipsoids drawn at the 30% probability level. The disorder is not shown.

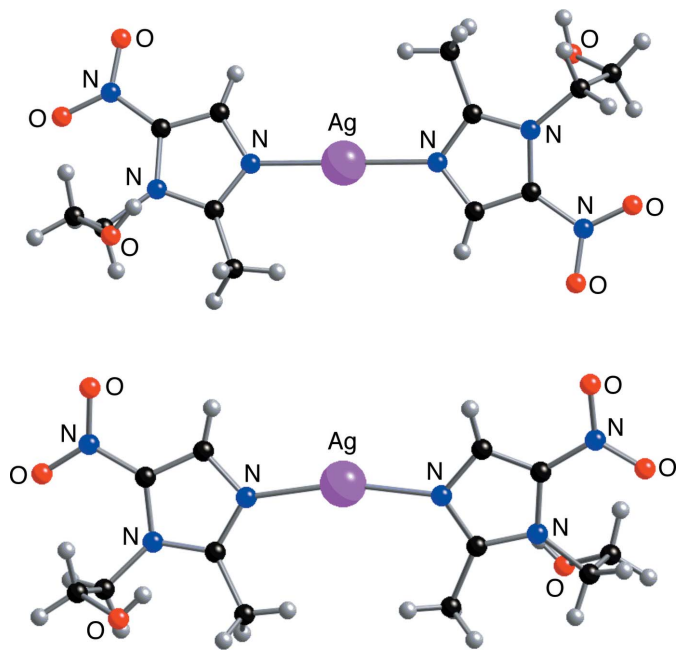


Figure 2
Comparison of the [Ag(MET)₂]⁺ units in [Ag(MET)₂]BF₄ (top) and [Ag(MET)₂]NO₃ (bottom).

A second interesting difference is that the N11–Ag–N21 angle of 175.7 (5)° for [Ag(MET)₂]BF₄ is much closer to 180° than is the corresponding value for [Ag(MET)₂]NO₃ [165.34 (4)°; Fun *et al.*, 2008]. It is possible that this could be attributed to the tetrafluoroborate ligand being considered a non-coordinating ion relative to nitrate, and this is reflected by the fact that [Ag(MET)₂]NO₃ exhibits Ag···O contacts of 2.63 and 2.67 Å, which are comparable to distances in other silver nitrate compounds (Wu *et al.*, 2012).

3. Supramolecular features

The hydroxyethyl group of one of the MET ligands [O21–H] serves as a donor in an intermolecular hydrogen-bonding interaction with the other hydroxyethyl group [O11–H] of an adjacent molecule. In turn, the latter hydroxyethyl group serves as a hydrogen-bond donor to a methanol molecule, which also hydrogen bonds to a tetrafluoroborate anion. In

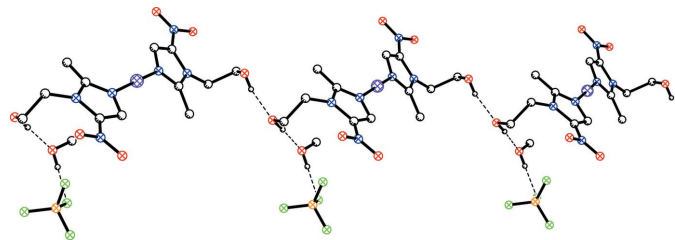


Figure 3
Part of a hydrogen-bonded chain along [001]. The disorder is not shown and hydrogen bonds are shown as dashed lines.

Table 1
Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| O1—H1 \cdots F2 | 0.84 | 1.84 | 2.673 (9) | 173 |
| O11—H11A \cdots O1 | 0.84 | 1.86 | 2.697 (10) | 175 |
| O21—H21A \cdots O11 ⁱ | 0.84 | 1.91 | 2.726 (11) | 164 |
| O21—H21A \cdots O11A ⁱ | 0.84 | 1.87 | 2.712 (11) | 176 |

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

the crystal, the components of the structure are linked into chains along [001] by the O—H \cdots O hydrogen bonds (Table 1 and Fig. 3).

4. Database survey

In addition to coordination to silver, metronidazole has also been shown to coordinate to other metals, and structurally characterized compounds have been reported for Co (Galván-Tejada *et al.*, 2002), Cu (Galván-Tejada *et al.*, 2002; Barba-Behrens *et al.*, 1991; Athar *et al.*, 2005; Ratajczak-Sitarz *et al.*, 1998; Bharti *et al.*, 2002), Zn (Galván-Tejada *et al.*, 2002), Ru (Wu *et al.*, 2003; Kennedy *et al.*, 2006), Rh (Dyson *et al.*, 1990; Nothenberg *et al.*, 1994), Pd (Bharti *et al.*, 2002; De Bondt *et al.*, 1994; Rochon *et al.*, 1993) and Pt (Bharti *et al.*, 2002; Bales *et al.*, 1983). In these compounds, the coordination number of the central atom ranges from four for Cu, Zn, Pd and Pt to six for Ru and Rh.

5. Synthesis and crystallization

Crystals of composition [Ag(MET)₂]BF₄·MeOH were obtained by combining AgBF₄ with MET in a 1:2 molar ratio in methanol and allowing the solution to evaporate slowly at room temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms were refined using a riding-model approximation with C—H = 0.95–0.99 Å, O—H = 0.84 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{O})$. One of the MET ligands was refined as rotationally disordered with occupancies of 0.501 (17) and 0.499 (17) and the configurations were modeled using the SAME command in *SHELXL2013* (Sheldrick, 2015). The tetrafluoroborate counter-ion was also refined as disordered and was modeled with two site occupancies, 0.539 (19) and 0.461 (19).

Acknowledgements

RKU would like to thank Pace University for research support. Gerard Parkin (Columbia University) is thanked for helpful discussions.

Table 2
Experimental details.

| | |
|--|--|
| Crystal data | |
| Chemical formula | [Ag(C ₆ H ₉ N ₃ O ₃) ₂]BF ₄ ·CH ₄ O |
| M_r | 569.04 |
| Crystal system, space group | Triclinic, $P\bar{1}$ |
| Temperature (K) | 130 |
| a, b, c (Å) | 9.2592 (10), 10.5339 (10), 12.3995 (12) |
| α, β, γ (°) | 106.940 (11), 92.788 (9), 112.439 (10) |
| V (Å ³) | 1051.7 (2) |
| Z | 2 |
| Radiation type | Mo $K\alpha$ |
| μ (mm ⁻¹) | 1.04 |
| Crystal size (mm) | 1.00 × 0.51 × 0.31 |
| Data collection | |
| Diffractometer | Bruker APEXII CCD |
| Absorption correction | Multi-scan (<i>SADABS</i> ; Bruker, 2013) |
| $T_{\text{min}}, T_{\text{max}}$ | 0.551, 0.747 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 17164, 6401, 6107 |
| R_{int} | 0.023 |
| $(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹) | 0.714 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)], wR(F^2), S$ | 0.028, 0.067, 1.12 |
| No. of reflections | 6401 |
| No. of parameters | 440 |
| No. of restraints | 144 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³) | 1.00, -1.03 |

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* and *SHELXL* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015).

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Acta Cryst. (2015). E71, 284-287 [doi:10.1107/S2056989015002819]

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Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINTE* (Bruker, 2013); data reduction: *SAINTE* (Bruker, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Bis[1-(2-hydroxyethyl)-2-methyl-5-nitro-1*H*-imidazole- κ N³]silver(I) tetrafluoridoborate methanol monosolvate

Crystal data

[Ag(C₆H₉N₃O₃)₂](BF₄)·CH₄O

M_r = 569.04

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

a = 9.2592 (10) Å

b = 10.5339 (10) Å

c = 12.3995 (12) Å

α = 106.940 (11)°

β = 92.788 (9)°

γ = 112.439 (10)°

V = 1051.7 (2) Å³

Z = 2

F(000) = 572

D_x = 1.797 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 9065 reflections

θ = 2.3–32.9°

μ = 1.04 mm⁻¹

T = 130 K

Block, colourless

1.00 × 0.51 × 0.31 mm

Data collection

Bruker APEXII CCD

diffractometer

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2013)

T_{min} = 0.551, *T_{max}* = 0.747

17164 measured reflections

6401 independent reflections

6107 reflections with *I* > 2 σ (*I*)

R_{int} = 0.023

θ_{\max} = 30.5°, θ_{\min} = 1.8°

h = -13→13

k = -15→15

l = -17→17

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2 σ (*F*²)] = 0.028

wR(*F*²) = 0.067

S = 1.12

6401 reflections

440 parameters

144 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[$\sigma^2(F_o^2) + (0.0161P)^2 + 1.0163P$]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.007

$\Delta\rho_{\max}$ = 1.00 e Å⁻³

$\Delta\rho_{\min}$ = -1.03 e Å⁻³

Extinction correction: *SHELXL2013* (Sheldrick, 2015), *F_c** = *kF_c*[1 + 0.001*xF_c*² $\lambda^3/\sin(2\theta)$]^{-1/4}

Extinction coefficient: 0.0027 (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.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|-------------|-------------|--------------|----------------------------------|------------|
| Ag | 0.34662 (2) | 0.82628 (2) | -0.00390 (2) | 0.02854 (5) | |
| N11 | 0.4710 (18) | 0.786 (2) | 0.1171 (7) | 0.018 (2) | 0.501 (17) |
| N12 | 0.5696 (15) | 0.7754 (15) | 0.2748 (8) | 0.0211 (16) | 0.501 (17) |
| N13 | 0.7178 (8) | 0.6262 (7) | 0.1956 (9) | 0.0273 (14) | 0.501 (17) |
| O11 | 0.3317 (11) | 0.6459 (13) | 0.4129 (9) | 0.0404 (16) | 0.501 (17) |
| H11A | 0.2863 | 0.5926 | 0.3453 | 0.048* | 0.501 (17) |
| O12 | 0.7349 (8) | 0.5491 (6) | 0.1064 (9) | 0.0406 (13) | 0.501 (17) |
| O13 | 0.7868 (7) | 0.6518 (5) | 0.2921 (10) | 0.0368 (14) | 0.501 (17) |
| C11 | 0.5471 (13) | 0.6968 (12) | 0.0851 (8) | 0.0227 (15) | 0.501 (17) |
| H11B | 0.5535 | 0.6475 | 0.0092 | 0.027* | 0.501 (17) |
| C12 | 0.6111 (12) | 0.6925 (11) | 0.1829 (8) | 0.0197 (15) | 0.501 (17) |
| C13 | 0.4855 (12) | 0.8352 (11) | 0.2301 (9) | 0.0149 (15) | 0.501 (17) |
| C14 | 0.3914 (18) | 0.9109 (16) | 0.2896 (10) | 0.039 (4) | 0.501 (17) |
| H14A | 0.3477 | 0.9470 | 0.2375 | 0.047* | 0.501 (17) |
| H14B | 0.4599 | 0.9932 | 0.3572 | 0.047* | 0.501 (17) |
| H14C | 0.3043 | 0.8428 | 0.3136 | 0.047* | 0.501 (17) |
| C15 | 0.5998 (12) | 0.7981 (8) | 0.3981 (7) | 0.0282 (14) | 0.501 (17) |
| H15A | 0.5816 | 0.8841 | 0.4416 | 0.034* | 0.501 (17) |
| H15B | 0.7124 | 0.8190 | 0.4220 | 0.034* | 0.501 (17) |
| C16 | 0.4946 (13) | 0.6673 (12) | 0.4271 (9) | 0.0346 (19) | 0.501 (17) |
| H16A | 0.5035 | 0.5792 | 0.3772 | 0.042* | 0.501 (17) |
| H16B | 0.5316 | 0.6806 | 0.5076 | 0.042* | 0.501 (17) |
| N11A | 0.470 (2) | 0.778 (2) | 0.1207 (9) | 0.028 (3) | 0.499 (17) |
| N12A | 0.5515 (16) | 0.7755 (15) | 0.2949 (8) | 0.0179 (12) | 0.499 (17) |
| N13A | 0.7308 (8) | 0.6471 (7) | 0.2389 (8) | 0.0224 (12) | 0.499 (17) |
| O11A | 0.2839 (10) | 0.6195 (10) | 0.4105 (9) | 0.0349 (13) | 0.499 (17) |
| H11C | 0.2496 | 0.5780 | 0.3398 | 0.042* | 0.499 (17) |
| O12A | 0.7814 (9) | 0.5918 (10) | 0.1564 (8) | 0.0368 (16) | 0.499 (17) |
| O13A | 0.7663 (7) | 0.6551 (5) | 0.3379 (7) | 0.0314 (11) | 0.499 (17) |
| C11A | 0.5764 (13) | 0.7137 (13) | 0.1118 (8) | 0.0212 (15) | 0.499 (17) |
| H11D | 0.6121 | 0.6788 | 0.0434 | 0.025* | 0.499 (17) |
| C12A | 0.6218 (12) | 0.7076 (11) | 0.2150 (7) | 0.0170 (13) | 0.499 (17) |
| C13A | 0.4578 (12) | 0.8126 (11) | 0.2331 (8) | 0.0140 (15) | 0.499 (17) |
| C14A | 0.3830 (17) | 0.9117 (14) | 0.2874 (9) | 0.033 (4) | 0.499 (17) |
| H14D | 0.4602 | 1.0132 | 0.3073 | 0.039* | 0.499 (17) |
| H14E | 0.3490 | 0.8932 | 0.3571 | 0.039* | 0.499 (17) |
| H14F | 0.2906 | 0.8940 | 0.2338 | 0.039* | 0.499 (17) |
| C15A | 0.5567 (9) | 0.7874 (8) | 0.4170 (6) | 0.0213 (11) | 0.499 (17) |
| H15C | 0.5264 | 0.8670 | 0.4571 | 0.026* | 0.499 (17) |

| | | | | | |
|------|--------------|--------------|---------------|-------------|------------|
| H15D | 0.6671 | 0.8140 | 0.4521 | 0.026* | 0.499 (17) |
| C16A | 0.4482 (11) | 0.6486 (10) | 0.4345 (9) | 0.0275 (16) | 0.499 (17) |
| H16C | 0.4661 | 0.5659 | 0.3840 | 0.033* | 0.499 (17) |
| H16D | 0.4758 | 0.6557 | 0.5148 | 0.033* | 0.499 (17) |
| N21 | 0.20394 (19) | 0.85591 (17) | -0.12498 (13) | 0.0280 (3) | |
| N22 | 0.06590 (19) | 0.82730 (17) | -0.28744 (13) | 0.0270 (3) | |
| N23 | -0.0132 (2) | 1.0346 (2) | -0.2186 (2) | 0.0433 (5) | |
| O21 | 0.25596 (17) | 0.86179 (17) | -0.45811 (12) | 0.0340 (3) | |
| H21A | 0.2608 | 0.7842 | -0.4975 | 0.051* | |
| O22 | -0.0234 (2) | 1.1214 (2) | -0.1326 (2) | 0.0737 (7) | |
| O23 | -0.0629 (3) | 1.0220 (3) | -0.3151 (2) | 0.0775 (8) | |
| C21 | 0.1481 (2) | 0.9618 (2) | -0.10391 (19) | 0.0350 (4) | |
| H21B | 0.1652 | 1.0344 | -0.0319 | 0.042* | |
| C22 | 0.0644 (2) | 0.94554 (19) | -0.20292 (19) | 0.0306 (4) | |
| C23 | 0.1525 (2) | 0.77595 (19) | -0.23615 (14) | 0.0250 (3) | |
| C24 | 0.1834 (3) | 0.6463 (2) | -0.29385 (16) | 0.0349 (4) | |
| H24A | 0.2455 | 0.6290 | -0.2379 | 0.042* | |
| H24B | 0.2429 | 0.6636 | -0.3552 | 0.042* | |
| H24C | 0.0821 | 0.5608 | -0.3265 | 0.042* | |
| C25 | -0.0068 (2) | 0.7652 (3) | -0.40921 (17) | 0.0375 (5) | |
| H25A | -0.1117 | 0.7691 | -0.4181 | 0.045* | |
| H25B | -0.0236 | 0.6617 | -0.4390 | 0.045* | |
| C26 | 0.0986 (3) | 0.8488 (3) | -0.4785 (2) | 0.0456 (6) | |
| H26A | 0.0535 | 0.7976 | -0.5612 | 0.055* | |
| H26B | 0.0997 | 0.9473 | -0.4580 | 0.055* | |
| C1 | 0.0949 (3) | 0.4974 (3) | 0.1155 (3) | 0.0598 (7) | |
| H1A | 0.0573 | 0.5721 | 0.1520 | 0.072* | |
| H1B | 0.0045 | 0.4081 | 0.0682 | 0.072* | |
| H1C | 0.1706 | 0.5328 | 0.0672 | 0.072* | |
| O1 | 0.1702 (3) | 0.4678 (3) | 0.20073 (19) | 0.0787 (8) | |
| H1 | 0.1856 | 0.3926 | 0.1706 | 0.094* | |
| B1 | 0.3719 (2) | 0.2457 (2) | 0.19180 (16) | 0.0263 (4) | |
| F1 | 0.4132 (11) | 0.1347 (10) | 0.1303 (10) | 0.0303 (13) | 0.461 (19) |
| F2 | 0.2428 (8) | 0.2394 (9) | 0.1193 (6) | 0.0313 (12) | 0.461 (19) |
| F3 | 0.4916 (8) | 0.3783 (7) | 0.2162 (11) | 0.0487 (18) | 0.461 (19) |
| F4 | 0.3177 (7) | 0.2190 (9) | 0.2882 (5) | 0.0395 (15) | 0.461 (19) |
| F1A | 0.4089 (12) | 0.1282 (10) | 0.1456 (10) | 0.045 (2) | 0.539 (19) |
| F2A | 0.2240 (8) | 0.2191 (10) | 0.1397 (9) | 0.0575 (19) | 0.539 (19) |
| F3A | 0.4837 (8) | 0.3671 (8) | 0.1725 (8) | 0.0457 (13) | 0.539 (19) |
| F4A | 0.3783 (17) | 0.2765 (12) | 0.3073 (4) | 0.080 (2) | 0.539 (19) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| Ag | 0.03598 (8) | 0.02979 (7) | 0.02076 (7) | 0.01318 (6) | 0.00356 (5) | 0.01063 (5) |
| N11 | 0.020 (4) | 0.016 (3) | 0.025 (4) | 0.013 (3) | 0.012 (3) | 0.008 (3) |
| N12 | 0.021 (3) | 0.022 (2) | 0.020 (3) | 0.012 (2) | -0.003 (2) | 0.002 (2) |
| N13 | 0.0202 (18) | 0.018 (2) | 0.045 (4) | 0.0087 (15) | 0.004 (3) | 0.012 (2) |

| | | | | | | |
|------|-------------|-------------|-------------|-------------|--------------|--------------|
| O11 | 0.040 (4) | 0.050 (5) | 0.0295 (18) | 0.023 (3) | 0.007 (3) | 0.004 (3) |
| O12 | 0.040 (2) | 0.034 (2) | 0.055 (4) | 0.0256 (18) | 0.018 (2) | 0.010 (2) |
| O13 | 0.0310 (19) | 0.0325 (17) | 0.051 (4) | 0.0180 (14) | -0.004 (2) | 0.015 (2) |
| C11 | 0.026 (4) | 0.020 (3) | 0.024 (3) | 0.010 (3) | 0.008 (3) | 0.010 (3) |
| C12 | 0.019 (2) | 0.015 (2) | 0.024 (4) | 0.0092 (18) | 0.004 (3) | 0.002 (3) |
| C13 | 0.007 (3) | 0.009 (3) | 0.025 (2) | -0.002 (3) | 0.0010 (18) | 0.0077 (17) |
| C14 | 0.054 (6) | 0.047 (7) | 0.028 (6) | 0.033 (5) | -0.004 (4) | 0.016 (5) |
| C15 | 0.035 (4) | 0.025 (2) | 0.024 (3) | 0.018 (3) | -0.005 (2) | 0.0024 (18) |
| C16 | 0.042 (5) | 0.047 (4) | 0.024 (3) | 0.028 (4) | 0.002 (3) | 0.011 (2) |
| N11A | 0.033 (5) | 0.027 (6) | 0.027 (5) | 0.011 (4) | 0.007 (3) | 0.014 (4) |
| N12A | 0.020 (2) | 0.0159 (18) | 0.019 (3) | 0.0107 (16) | 0.0043 (19) | 0.004 (2) |
| N13A | 0.019 (2) | 0.020 (2) | 0.032 (3) | 0.0085 (16) | 0.006 (2) | 0.012 (2) |
| O11A | 0.031 (3) | 0.036 (3) | 0.0304 (18) | 0.008 (2) | 0.010 (3) | 0.0074 (17) |
| O12A | 0.041 (3) | 0.049 (3) | 0.050 (3) | 0.035 (3) | 0.030 (3) | 0.032 (3) |
| O13A | 0.034 (2) | 0.0296 (15) | 0.033 (3) | 0.0163 (13) | -0.0055 (17) | 0.0111 (16) |
| C11A | 0.025 (4) | 0.024 (3) | 0.021 (3) | 0.015 (3) | 0.009 (3) | 0.010 (3) |
| C12A | 0.0166 (19) | 0.018 (3) | 0.016 (3) | 0.0060 (19) | 0.001 (2) | 0.007 (3) |
| C13A | 0.006 (3) | 0.008 (3) | 0.022 (2) | -0.004 (3) | 0.0011 (17) | 0.0072 (17) |
| C14A | 0.057 (6) | 0.042 (6) | 0.023 (5) | 0.045 (6) | 0.018 (4) | 0.009 (4) |
| C15A | 0.024 (3) | 0.0262 (19) | 0.015 (2) | 0.012 (2) | 0.0007 (16) | 0.0060 (14) |
| C16A | 0.037 (4) | 0.025 (2) | 0.022 (2) | 0.012 (3) | 0.009 (3) | 0.0104 (16) |
| N21 | 0.0342 (8) | 0.0243 (7) | 0.0263 (7) | 0.0143 (6) | 0.0021 (6) | 0.0072 (6) |
| N22 | 0.0333 (8) | 0.0331 (8) | 0.0280 (7) | 0.0192 (6) | 0.0108 (6) | 0.0207 (6) |
| N23 | 0.0287 (8) | 0.0314 (9) | 0.0875 (16) | 0.0166 (7) | 0.0213 (9) | 0.0373 (10) |
| O21 | 0.0335 (7) | 0.0498 (8) | 0.0312 (7) | 0.0258 (6) | 0.0119 (5) | 0.0185 (6) |
| O22 | 0.0565 (11) | 0.0321 (9) | 0.1166 (19) | 0.0296 (9) | -0.0178 (12) | -0.0075 (10) |
| O23 | 0.1030 (17) | 0.131 (2) | 0.0963 (16) | 0.1008 (17) | 0.0702 (14) | 0.0957 (17) |
| C21 | 0.0299 (9) | 0.0222 (8) | 0.0454 (11) | 0.0116 (7) | 0.0000 (8) | 0.0007 (7) |
| C22 | 0.0243 (8) | 0.0201 (7) | 0.0522 (11) | 0.0102 (6) | 0.0080 (7) | 0.0173 (8) |
| C23 | 0.0348 (9) | 0.0254 (8) | 0.0225 (7) | 0.0163 (7) | 0.0063 (6) | 0.0135 (6) |
| C24 | 0.0541 (12) | 0.0358 (10) | 0.0248 (8) | 0.0301 (9) | 0.0044 (8) | 0.0089 (7) |
| C25 | 0.0317 (9) | 0.0647 (14) | 0.0302 (9) | 0.0243 (9) | 0.0083 (7) | 0.0295 (9) |
| C26 | 0.0383 (11) | 0.0888 (18) | 0.0413 (11) | 0.0383 (12) | 0.0188 (9) | 0.0481 (13) |
| C1 | 0.0312 (11) | 0.0628 (17) | 0.0691 (18) | 0.0079 (11) | 0.0019 (11) | 0.0160 (14) |
| O1 | 0.0709 (13) | 0.0848 (15) | 0.0653 (13) | 0.0613 (13) | -0.0276 (11) | -0.0290 (11) |
| B1 | 0.0270 (9) | 0.0233 (8) | 0.0235 (8) | 0.0066 (7) | 0.0073 (7) | 0.0053 (7) |
| F1 | 0.037 (3) | 0.028 (2) | 0.031 (2) | 0.0183 (18) | 0.0130 (18) | 0.0090 (19) |
| F2 | 0.037 (3) | 0.029 (2) | 0.0255 (16) | 0.015 (2) | 0.0004 (15) | 0.0061 (13) |
| F3 | 0.0250 (17) | 0.0217 (15) | 0.083 (5) | 0.0004 (12) | 0.007 (3) | 0.007 (3) |
| F4 | 0.039 (2) | 0.055 (3) | 0.0244 (17) | 0.0204 (19) | 0.0125 (14) | 0.0110 (17) |
| F1A | 0.070 (4) | 0.031 (2) | 0.039 (3) | 0.025 (2) | 0.010 (2) | 0.0146 (19) |
| F2A | 0.0192 (14) | 0.047 (3) | 0.074 (4) | 0.0064 (16) | -0.001 (2) | -0.015 (3) |
| F3A | 0.0341 (16) | 0.0302 (19) | 0.068 (3) | 0.0038 (13) | 0.006 (2) | 0.023 (2) |
| F4A | 0.125 (6) | 0.081 (4) | 0.0201 (16) | 0.033 (5) | 0.019 (2) | 0.008 (2) |

Geometric parameters (Å, °)

| | | | |
|-------------|-------------|----------------|-----------|
| Ag—N11 | 2.082 (15) | C15A—C16A | 1.510 (9) |
| Ag—N21 | 2.1193 (15) | C15A—H15C | 0.9900 |
| Ag—N11A | 2.163 (16) | C15A—H15D | 0.9900 |
| N11—C13 | 1.325 (9) | C16A—H16C | 0.9900 |
| N11—C11 | 1.365 (9) | C16A—H16D | 0.9900 |
| N12—C13 | 1.356 (9) | N21—C23 | 1.338 (2) |
| N12—C12 | 1.389 (9) | N21—C21 | 1.365 (2) |
| N12—C15 | 1.467 (8) | N22—C23 | 1.350 (2) |
| N13—O12 | 1.224 (6) | N22—C22 | 1.380 (3) |
| N13—O13 | 1.233 (6) | N22—C25 | 1.466 (3) |
| N13—C12 | 1.436 (8) | N23—O23 | 1.212 (3) |
| O11—C16 | 1.431 (9) | N23—O22 | 1.221 (3) |
| O11—H11A | 0.8400 | N23—C22 | 1.429 (2) |
| C11—C12 | 1.345 (9) | O21—C26 | 1.413 (2) |
| C11—H11B | 0.9500 | O21—H21A | 0.8400 |
| C13—C14 | 1.479 (9) | C21—C22 | 1.349 (3) |
| C14—H14A | 0.9800 | C21—H21B | 0.9500 |
| C14—H14B | 0.9800 | C23—C24 | 1.486 (2) |
| C14—H14C | 0.9800 | C24—H24A | 0.9800 |
| C15—C16 | 1.509 (9) | C24—H24B | 0.9800 |
| C15—H15A | 0.9900 | C24—H24C | 0.9800 |
| C15—H15B | 0.9900 | C25—C26 | 1.527 (3) |
| C16—H16A | 0.9900 | C25—H25A | 0.9900 |
| C16—H16B | 0.9900 | C25—H25B | 0.9900 |
| N11A—C13A | 1.365 (8) | C26—H26A | 0.9900 |
| N11A—C11A | 1.379 (9) | C26—H26B | 0.9900 |
| N12A—C13A | 1.369 (9) | C1—O1 | 1.410 (4) |
| N12A—C12A | 1.389 (9) | C1—H1A | 0.9800 |
| N12A—C15A | 1.483 (8) | C1—H1B | 0.9800 |
| N13A—O13A | 1.226 (5) | C1—H1C | 0.9800 |
| N13A—O12A | 1.231 (6) | O1—H1 | 0.8400 |
| N13A—C12A | 1.445 (8) | B1—F3 | 1.346 (6) |
| O11A—C16A | 1.430 (9) | B1—F4A | 1.366 (4) |
| O11A—H11C | 0.8400 | B1—F2A | 1.373 (6) |
| C11A—C12A | 1.355 (9) | B1—F4 | 1.381 (4) |
| C11A—H11D | 0.9500 | B1—F1A | 1.382 (7) |
| C13A—C14A | 1.491 (9) | B1—F1 | 1.395 (6) |
| C14A—H14D | 0.9800 | B1—F3A | 1.399 (5) |
| C14A—H14E | 0.9800 | B1—F2 | 1.428 (6) |
| C14A—H14F | 0.9800 | | |
| N11—Ag—N21 | 175.7 (5) | N12A—C15A—H15D | 108.9 |
| C13—N11—C11 | 111.5 (7) | C16A—C15A—H15D | 108.9 |
| C13—N11—Ag | 128.4 (6) | H15C—C15A—H15D | 107.7 |
| C11—N11—Ag | 121.4 (7) | O11A—C16A—C15A | 112.9 (7) |
| C13—N12—C12 | 106.6 (7) | O11A—C16A—H16C | 109.0 |

| | | | |
|----------------|------------|----------------|-------------|
| C13—N12—C15 | 122.1 (7) | C15A—C16A—H16C | 109.0 |
| C12—N12—C15 | 131.2 (7) | O11A—C16A—H16D | 109.0 |
| O12—N13—O13 | 125.0 (6) | C15A—C16A—H16D | 109.0 |
| O12—N13—C12 | 115.7 (5) | H16C—C16A—H16D | 107.8 |
| O13—N13—C12 | 119.2 (5) | C23—N21—C21 | 106.99 (16) |
| C16—O11—H11A | 109.5 | C23—N21—Ag | 126.88 (12) |
| C12—C11—N11 | 105.1 (7) | C21—N21—Ag | 126.14 (13) |
| C12—C11—H11B | 127.5 | C23—N22—C22 | 105.84 (15) |
| N11—C11—H11B | 127.5 | C23—N22—C25 | 124.53 (16) |
| C11—C12—N12 | 109.3 (7) | C22—N22—C25 | 129.62 (16) |
| C11—C12—N13 | 127.3 (6) | O23—N23—O22 | 123.6 (2) |
| N12—C12—N13 | 123.2 (6) | O23—N23—C22 | 119.0 (2) |
| N11—C13—N12 | 107.3 (7) | O22—N23—C22 | 117.3 (2) |
| N11—C13—C14 | 122.3 (8) | C26—O21—H21A | 109.5 |
| N12—C13—C14 | 127.2 (10) | C22—C21—N21 | 108.23 (17) |
| C13—C14—H14A | 109.5 | C22—C21—H21B | 125.9 |
| C13—C14—H14B | 109.5 | N21—C21—H21B | 125.9 |
| H14A—C14—H14B | 109.5 | C21—C22—N22 | 108.31 (15) |
| C13—C14—H14C | 109.5 | C21—C22—N23 | 126.3 (2) |
| H14A—C14—H14C | 109.5 | N22—C22—N23 | 125.40 (19) |
| H14B—C14—H14C | 109.5 | N21—C23—N22 | 110.63 (15) |
| N12—C15—C16 | 112.0 (8) | N21—C23—C24 | 124.38 (15) |
| N12—C15—H15A | 109.2 | N22—C23—C24 | 124.97 (16) |
| C16—C15—H15A | 109.2 | C23—C24—H24A | 109.5 |
| N12—C15—H15B | 109.2 | C23—C24—H24B | 109.5 |
| C16—C15—H15B | 109.2 | H24A—C24—H24B | 109.5 |
| H15A—C15—H15B | 107.9 | C23—C24—H24C | 109.5 |
| O11—C16—C15 | 112.1 (8) | H24A—C24—H24C | 109.5 |
| O11—C16—H16A | 109.2 | H24B—C24—H24C | 109.5 |
| C15—C16—H16A | 109.2 | N22—C25—C26 | 110.98 (19) |
| O11—C16—H16B | 109.2 | N22—C25—H25A | 109.4 |
| C15—C16—H16B | 109.2 | C26—C25—H25A | 109.4 |
| H16A—C16—H16B | 107.9 | N22—C25—H25B | 109.4 |
| C13A—N11A—C11A | 103.4 (6) | C26—C25—H25B | 109.4 |
| C13A—N11A—Ag | 122.6 (6) | H25A—C25—H25B | 108.0 |
| C11A—N11A—Ag | 132.1 (8) | O21—C26—C25 | 111.84 (15) |
| C13A—N12A—C12A | 104.6 (7) | O21—C26—H26A | 109.2 |
| C13A—N12A—C15A | 127.3 (7) | C25—C26—H26A | 109.2 |
| C12A—N12A—C15A | 127.5 (7) | O21—C26—H26B | 109.2 |
| O13A—N13A—O12A | 125.3 (5) | C25—C26—H26B | 109.2 |
| O13A—N13A—C12A | 118.6 (5) | H26A—C26—H26B | 107.9 |
| O12A—N13A—C12A | 116.1 (5) | O1—C1—H1A | 109.5 |
| C16A—O11A—H11C | 109.5 | O1—C1—H1B | 109.5 |
| C12A—C11A—N11A | 110.0 (10) | H1A—C1—H1B | 109.5 |
| C12A—C11A—H11D | 124.5 | O1—C1—H1C | 109.5 |
| N11A—C11A—H11D | 124.5 | H1A—C1—H1C | 109.5 |
| C11A—C12A—N12A | 107.9 (7) | H1B—C1—H1C | 109.5 |
| C11A—C12A—N13A | 125.8 (7) | C1—O1—H1 | 109.5 |

| | | | |
|---------------------|-------------|---------------------|--------------|
| N12A—C12A—N13A | 126.2 (7) | F4A—B1—F2A | 110.2 (4) |
| N11A—C13A—N12A | 112.9 (7) | F3—B1—F4 | 112.8 (4) |
| N11A—C13A—C14A | 123.6 (8) | F4A—B1—F1A | 110.7 (5) |
| N12A—C13A—C14A | 122.9 (9) | F2A—B1—F1A | 110.7 (5) |
| C13A—C14A—H14D | 109.5 | F3—B1—F1 | 111.9 (5) |
| C13A—C14A—H14E | 109.5 | F4—B1—F1 | 109.9 (6) |
| H14D—C14A—H14E | 109.5 | F4A—B1—F3A | 108.5 (4) |
| C13A—C14A—H14F | 109.5 | F2A—B1—F3A | 108.4 (5) |
| H14D—C14A—H14F | 109.5 | F1A—B1—F3A | 108.4 (5) |
| H14E—C14A—H14F | 109.5 | F3—B1—F2 | 107.7 (5) |
| N12A—C15A—C16A | 113.4 (8) | F4—B1—F2 | 107.7 (3) |
| N12A—C15A—H15C | 108.9 | F1—B1—F2 | 106.5 (5) |
| C16A—C15A—H15C | 108.9 | | |
| | | | |
| C13—N11—C11—C12 | 1.2 (14) | Ag—N11A—C13A—N12A | -178.7 (9) |
| Ag—N11—C11—C12 | -179.8 (8) | C11A—N11A—C13A—C14A | 166.5 (12) |
| N11—C11—C12—N12 | 1.2 (14) | Ag—N11A—C13A—C14A | -12 (2) |
| N11—C11—C12—N13 | -173.5 (11) | C12A—N12A—C13A—N11A | -4.3 (15) |
| C13—N12—C12—C11 | -3.1 (15) | C15A—N12A—C13A—N11A | -176.3 (12) |
| C15—N12—C12—C11 | 175.3 (13) | C12A—N12A—C13A—C14A | -168.1 (12) |
| C13—N12—C12—N13 | 171.8 (10) | C15A—N12A—C13A—C14A | 20 (2) |
| C15—N12—C12—N13 | -10 (2) | C13A—N12A—C15A—C16A | 93.2 (15) |
| O12—N13—C12—C11 | -10.3 (15) | C12A—N12A—C15A—C16A | -77.0 (15) |
| O13—N13—C12—C11 | 168.2 (11) | N12A—C15A—C16A—O11A | -71.9 (10) |
| O12—N13—C12—N12 | 175.6 (11) | C23—N21—C21—C22 | -0.5 (2) |
| O13—N13—C12—N12 | -5.8 (16) | Ag—N21—C21—C22 | 179.02 (13) |
| C11—N11—C13—N12 | -3.2 (14) | N21—C21—C22—N22 | 0.6 (2) |
| Ag—N11—C13—N12 | 178.0 (9) | N21—C21—C22—N23 | -179.57 (17) |
| C11—N11—C13—C14 | -168.5 (12) | C23—N22—C22—C21 | -0.4 (2) |
| Ag—N11—C13—C14 | 10 (2) | C25—N22—C22—C21 | -179.75 (19) |
| C12—N12—C13—N11 | 3.8 (14) | C23—N22—C22—N23 | 179.73 (17) |
| C15—N12—C13—N11 | -174.8 (11) | C25—N22—C22—N23 | 0.4 (3) |
| C12—N12—C13—C14 | 167.9 (13) | O23—N23—C22—C21 | 168.6 (2) |
| C15—N12—C13—C14 | -11 (2) | O22—N23—C22—C21 | -10.3 (3) |
| C13—N12—C15—C16 | 103.4 (13) | O23—N23—C22—N22 | -11.6 (3) |
| C12—N12—C15—C16 | -74.8 (16) | O22—N23—C22—N22 | 169.5 (2) |
| N12—C15—C16—O11 | -68.9 (10) | C21—N21—C23—N22 | 0.2 (2) |
| C13A—N11A—C11A—C12A | 1.5 (18) | Ag—N21—C23—N22 | -179.28 (12) |
| Ag—N11A—C11A—C12A | -179.9 (13) | C21—N21—C23—C24 | -178.24 (19) |
| N11A—C11A—C12A—N12A | -2.1 (15) | Ag—N21—C23—C24 | 2.2 (3) |
| N11A—C11A—C12A—N13A | -179.3 (10) | C22—N22—C23—N21 | 0.1 (2) |
| C13A—N12A—C12A—C11A | 3.7 (15) | C25—N22—C23—N21 | 179.47 (17) |
| C15A—N12A—C12A—C11A | 175.8 (12) | C22—N22—C23—C24 | 178.57 (18) |
| C13A—N12A—C12A—N13A | -179.0 (10) | C25—N22—C23—C24 | -2.1 (3) |
| C15A—N12A—C12A—N13A | -7 (2) | C23—N22—C25—C26 | -96.7 (2) |
| O13A—N13A—C12A—C11A | 176.7 (10) | C22—N22—C25—C26 | 82.5 (2) |
| O12A—N13A—C12A—C11A | -2.9 (15) | N22—C25—C26—O21 | 52.0 (3) |
| O13A—N13A—C12A—N12A | -0.1 (16) | C13—N11—N21—C23 | -160.8 (9) |

| | | | |
|---------------------|-------------|-------------------|-------------|
| O12A—N13A—C12A—N12A | -179.7 (11) | C13A—N11A—N21—C23 | -148.6 (11) |
| C11A—N11A—C13A—N12A | 0.8 (18) | | |

Hydrogen-bond geometry (Å, °)

| <i>D—H...A</i> | <i>D—H</i> | <i>H...A</i> | <i>D...A</i> | <i>D—H...A</i> |
|------------------------------|------------|--------------|--------------|----------------|
| O1—H1...F2 | 0.84 | 1.84 | 2.673 (9) | 173 |
| O11—H11A...O1 | 0.84 | 1.86 | 2.697 (10) | 175 |
| O21—H21A...O11 ⁱ | 0.84 | 1.91 | 2.726 (11) | 164 |
| O21—H21A...O11A ⁱ | 0.84 | 1.87 | 2.712 (11) | 176 |

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