

Bis[μ -2,3-bis(imidazol-1-ylmethyl)-quinoxaline]disilver(I) bis(tetrafluoroborate)

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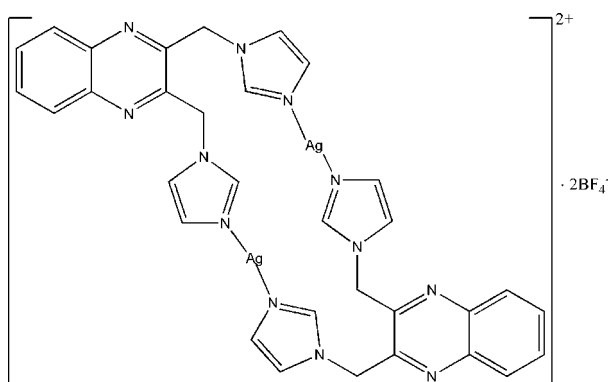
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.046; wR factor = 0.108; data-to-parameter ratio = 14.1.

The title compound, $[\text{Ag}_2(\text{C}_{16}\text{H}_{14}\text{N}_6)_2](\text{BF}_4)_2$, forms a centrosymmetric 22-membered metallamacrocycle *via* two Ag^{I} ions bridging two 2,3-bis(imidazol-1-ylmethyl)quinoxaline ligands. The Ag^{I} ions are coordinated by two N donors of the imidazole groups, forming an approximately linear coordination geometry.

Related literature

For related literature, see: Li, Liu *et al.* (2007); Li, Tao *et al.* (2007); Zhang *et al.* (2006); Zou *et al.* (2004).



Experimental

Crystal data

$[\text{Ag}_2(\text{C}_{16}\text{H}_{14}\text{N}_6)_2](\text{BF}_4)_2$	$\gamma = 73.208$ (4) $^\circ$
$M_r = 970.02$	$V = 881.4$ (4) Å ³
Triclinic, $P\bar{1}$	$Z = 1$
$a = 8.5398$ (19) Å	Mo $K\alpha$ radiation
$b = 9.311$ (2) Å	$\mu = 1.20$ mm ⁻¹
$c = 12.104$ (3) Å	$T = 293$ (2) K
$\alpha = 76.350$ (4) $^\circ$	$0.20 \times 0.18 \times 0.16$ mm
$\beta = 76.603$ (4) $^\circ$	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	5006 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 1998)	3556 independent reflections
$T_{\text{min}} = 0.796$, $T_{\text{max}} = 0.831$	2326 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$	253 parameters
$wR(F^2) = 0.108$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.61$ e Å ⁻³
3556 reflections	$\Delta\rho_{\text{min}} = -0.47$ e Å ⁻³

Table 1

 Selected geometric parameters (Å, $^\circ$).

Ag1—N1	2.105 (4)	Ag1—N6 ⁱ	2.112 (4)
N1—Ag1—N6 ⁱ	178.55 (16)		

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2577).

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supplementary materials

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Bis[μ -2,3-bis(imidazol-1-ylmethyl)quinoxaline]disilver(I) bis(tetrafluoridoborate)

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Comment

The synthesis of metal-organic macrocyclic and polymeric compounds have drawn much attention in recent years not only because of their interesting properties and potential applications, but also due to variety of structural topologies (Zou, *et al.*, 2004; Zhang *et al.*, 2006; Li Tao *et al.*, 2007). Much progress has been reported in the study of crystal engineering of supramolecular architectures using N-donor ligands (Li, Liu *et al.*, 2007). The title compound (I) is a dinuclear Ag(I) complex formed with two 2,3-bis(imidazol-1-ylmethyl)quinoxaline ligands (Fig.1). The Ag^I atoms are coordinated by two N donors of the imiazole groups, forming a approximately linear coordination geometry. The molecular structure forms a centrosymmetric 22-membered metallacrocyclic.

Experimental

2,3-bis(imidazol-1-ylmethyl)quinoxaline was synthesized by a modified literature method (Li, Liu *et al.*, 2007). A solution of 2,3-bis(imidazol-1-ylmethyl)quinoxaline (32 mg, 0.1 mmol) in MeOH (10 ml) was carefully layered on top of a AgBF₄ (20 mg, 0.1 mmol) solution in H₂O in a test-tube, which was placed in the darkness. After 10 d at room temperature, colorless single crystals of (I) were obtained (yield: 30 mg, 30%).

Refinement

The H atoms were placed in calculated positions and treated in the subsequent refinement as riding atoms, with C—H = 0.93 and 0.97 Å; $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}$.

Figures

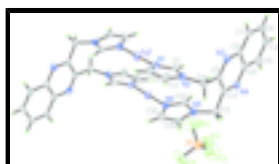


Fig. 1. the molecular with 30% displacement probability. Unlabelled atoms are generated by the symmetry operation ($-x, -y, -z$). Only the symmetry unique BF₄ anion is shown.

Bis[μ -2,3-bis(imidazol-1-ylmethyl)quinoxaline]disilver(I) bis(tetrafluoridoborate)

Crystal data

[Ag₂(C₁₆H₁₄N₆)₂](BF₄)₂

$M_r = 970.02$

Triclinic, $P\bar{1}$

Hall symbol: $-P 1$

$Z = 1$

$F_{000} = 480$

$D_x = 1.827 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

supplementary materials

$a = 8.5398 (19) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 9.311 (2) \text{ \AA}$	Cell parameters from 1538 reflections
$c = 12.104 (3) \text{ \AA}$	$\theta = 2.3\text{--}23.9^\circ$
$\alpha = 76.350 (4)^\circ$	$\mu = 1.20 \text{ mm}^{-1}$
$\beta = 76.603 (4)^\circ$	$T = 293 (2) \text{ K}$
$\gamma = 73.208 (4)^\circ$	Block, colorless
$V = 881.4 (4) \text{ \AA}^3$	$0.20 \times 0.18 \times 0.16 \text{ mm}$

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3556 independent reflections
Radiation source: fine-focus sealed tube	2326 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.023$
$T = 293(2) \text{ K}$	$\theta_{\text{max}} = 26.4^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.8^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.796$, $T_{\text{max}} = 0.831$	$k = -11 \rightarrow 10$
5006 measured reflections	$l = -11 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.108$	$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 1.1091P]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
3556 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
253 parameters	$\Delta\rho_{\text{max}} = 0.61 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.47 \text{ e \AA}^{-3}$
	Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ag1	0.19306 (5)	-0.02219 (5)	-0.09278 (4)	0.05500 (18)
N1	0.2915 (5)	0.1119 (5)	-0.0225 (3)	0.0468 (11)
N2	0.3706 (5)	0.2045 (4)	0.1003 (3)	0.0396 (10)
N3	0.5399 (5)	-0.0159 (5)	0.3042 (3)	0.0401 (10)
N4	0.2569 (5)	-0.0400 (5)	0.4717 (3)	0.0405 (10)
N5	0.0162 (4)	0.2173 (4)	0.2876 (3)	0.0375 (9)
N6	-0.0997 (5)	0.1574 (5)	0.1666 (3)	0.0406 (10)
C1	0.3174 (6)	0.0900 (6)	0.0840 (4)	0.0386 (11)
H1A	0.3010	0.0061	0.1406	0.046*
C2	0.3313 (8)	0.2453 (7)	-0.0763 (5)	0.0663 (17)
H2A	0.3255	0.2896	-0.1531	0.080*
C3	0.3806 (8)	0.3042 (7)	-0.0024 (5)	0.0647 (17)
H3A	0.4146	0.3944	-0.0181	0.078*
C4	0.4059 (6)	0.2277 (6)	0.2072 (4)	0.0429 (12)
H4A	0.3248	0.3162	0.2323	0.051*
H4B	0.5149	0.2479	0.1919	0.051*
C5	0.4007 (6)	0.0904 (5)	0.3025 (4)	0.0368 (11)
C6	0.5423 (6)	-0.1396 (5)	0.3899 (4)	0.0385 (12)
C7	0.6913 (6)	-0.2570 (6)	0.3952 (5)	0.0484 (13)
H7A	0.7858	-0.2494	0.3399	0.058*
C8	0.6947 (7)	-0.3806 (6)	0.4817 (5)	0.0511 (14)
H8A	0.7916	-0.4584	0.4842	0.061*
C9	0.5553 (7)	-0.3927 (6)	0.5666 (5)	0.0526 (15)
H9A	0.5607	-0.4773	0.6259	0.063*
C10	0.4105 (7)	-0.2807 (6)	0.5633 (5)	0.0496 (14)
H10A	0.3177	-0.2901	0.6198	0.060*
C11	0.4018 (6)	-0.1520 (5)	0.4748 (4)	0.0398 (12)
C12	0.2574 (6)	0.0785 (5)	0.3867 (4)	0.0366 (11)
C13	0.0938 (6)	0.2008 (6)	0.3887 (4)	0.0444 (13)
H13A	0.0175	0.1747	0.4588	0.053*
H13B	0.1138	0.2977	0.3900	0.053*
C14	-0.0340 (6)	0.1069 (5)	0.2612 (4)	0.0393 (11)
H14A	-0.0237	0.0085	0.3039	0.047*
C15	-0.0222 (6)	0.3462 (6)	0.2053 (5)	0.0490 (13)
H15A	-0.0029	0.4408	0.2011	0.059*
C16	-0.0935 (6)	0.3070 (6)	0.1324 (5)	0.0512 (14)
H16A	-0.1327	0.3720	0.0681	0.061*
B1	0.1799 (10)	0.6832 (8)	0.2227 (7)	0.0602 (19)
F1	0.2917 (7)	0.5925 (6)	0.1565 (6)	0.162 (2)
F2	0.2537 (7)	0.7599 (6)	0.2651 (5)	0.1363 (19)
F3	0.1090 (6)	0.5843 (5)	0.3089 (4)	0.1135 (15)
F4	0.0645 (7)	0.7784 (6)	0.1667 (5)	0.158 (2)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0564 (3)	0.0600 (3)	0.0593 (3)	-0.0085 (2)	-0.0270 (2)	-0.0223 (2)
N1	0.052 (3)	0.053 (3)	0.039 (2)	-0.010 (2)	-0.013 (2)	-0.012 (2)
N2	0.046 (2)	0.037 (2)	0.041 (2)	-0.0140 (19)	-0.0130 (19)	-0.0062 (19)
N3	0.040 (2)	0.045 (3)	0.042 (2)	-0.011 (2)	-0.0138 (19)	-0.013 (2)
N4	0.045 (2)	0.043 (2)	0.036 (2)	-0.006 (2)	-0.0160 (19)	-0.0107 (19)
N5	0.036 (2)	0.037 (2)	0.043 (2)	-0.0073 (18)	-0.0128 (18)	-0.0086 (19)
N6	0.038 (2)	0.045 (3)	0.042 (2)	-0.009 (2)	-0.0150 (19)	-0.0063 (19)
C1	0.041 (3)	0.039 (3)	0.037 (3)	-0.013 (2)	-0.006 (2)	-0.007 (2)
C2	0.102 (5)	0.068 (4)	0.037 (3)	-0.034 (4)	-0.023 (3)	0.004 (3)
C3	0.100 (5)	0.058 (4)	0.044 (3)	-0.044 (4)	-0.016 (3)	0.009 (3)
C4	0.047 (3)	0.041 (3)	0.048 (3)	-0.016 (2)	-0.012 (2)	-0.013 (2)
C5	0.040 (3)	0.035 (3)	0.041 (3)	-0.010 (2)	-0.020 (2)	-0.006 (2)
C6	0.042 (3)	0.038 (3)	0.041 (3)	-0.004 (2)	-0.019 (2)	-0.014 (2)
C7	0.044 (3)	0.050 (3)	0.053 (3)	-0.002 (3)	-0.018 (3)	-0.015 (3)
C8	0.053 (3)	0.038 (3)	0.069 (4)	0.004 (3)	-0.031 (3)	-0.020 (3)
C9	0.070 (4)	0.036 (3)	0.057 (3)	-0.006 (3)	-0.035 (3)	-0.005 (2)
C10	0.054 (3)	0.047 (3)	0.049 (3)	-0.006 (3)	-0.019 (3)	-0.010 (3)
C11	0.043 (3)	0.041 (3)	0.041 (3)	0.000 (2)	-0.020 (2)	-0.019 (2)
C12	0.040 (3)	0.039 (3)	0.037 (3)	-0.007 (2)	-0.017 (2)	-0.014 (2)
C13	0.043 (3)	0.046 (3)	0.047 (3)	0.000 (2)	-0.018 (2)	-0.018 (2)
C14	0.040 (3)	0.034 (3)	0.042 (3)	-0.004 (2)	-0.010 (2)	-0.006 (2)
C15	0.054 (3)	0.031 (3)	0.063 (4)	-0.006 (2)	-0.027 (3)	0.000 (3)
C16	0.056 (3)	0.043 (3)	0.055 (3)	-0.009 (3)	-0.031 (3)	0.006 (3)
B1	0.074 (5)	0.035 (4)	0.072 (5)	-0.012 (4)	-0.025 (4)	-0.002 (3)
F1	0.146 (5)	0.094 (4)	0.223 (6)	-0.029 (3)	0.056 (4)	-0.072 (4)
F2	0.184 (5)	0.103 (4)	0.162 (5)	-0.071 (4)	-0.066 (4)	-0.022 (3)
F3	0.114 (3)	0.109 (4)	0.120 (4)	-0.053 (3)	-0.038 (3)	0.024 (3)
F4	0.170 (5)	0.121 (4)	0.154 (5)	0.030 (4)	-0.099 (4)	0.016 (3)

Geometric parameters (\AA , $^\circ$)

Ag1—N1	2.105 (4)	C4—H4B	0.9700
Ag1—N6 ⁱ	2.112 (4)	C5—C12	1.413 (7)
Ag1—Ag1 ⁱ	3.5081 (11)	C6—C11	1.400 (7)
N1—C1	1.317 (5)	C6—C7	1.421 (7)
N1—C2	1.357 (7)	C7—C8	1.359 (8)
N2—C1	1.342 (6)	C7—H7A	0.9300
N2—C3	1.367 (7)	C8—C9	1.393 (8)
N2—C4	1.471 (5)	C8—H8A	0.9300
N3—C5	1.309 (6)	C9—C10	1.370 (7)
N3—C6	1.355 (6)	C9—H9A	0.9300
N4—C12	1.319 (6)	C10—C11	1.403 (7)
N4—C11	1.370 (6)	C10—H10A	0.9300
N5—C14	1.348 (6)	C12—C13	1.525 (6)

N5—C15	1.378 (6)	C13—H13A	0.9700
N5—C13	1.479 (5)	C13—H13B	0.9700
N6—C14	1.319 (5)	C14—H14A	0.9300
N6—C16	1.369 (6)	C15—C16	1.346 (7)
N6—Ag1 ⁱ	2.112 (4)	C15—H15A	0.9300
C1—H1A	0.9300	C16—H16A	0.9300
C2—C3	1.348 (7)	B1—F4	1.318 (8)
C2—H2A	0.9300	B1—F2	1.324 (8)
C3—H3A	0.9300	B1—F1	1.328 (8)
C4—C5	1.510 (7)	B1—F3	1.370 (8)
C4—H4A	0.9700		
N1—Ag1—N6 ⁱ	178.55 (16)	C8—C7—H7A	120.2
N1—Ag1—Ag1 ⁱ	97.02 (11)	C6—C7—H7A	120.2
N6 ⁱ —Ag1—Ag1 ⁱ	84.42 (11)	C7—C8—C9	121.1 (5)
C1—N1—C2	105.7 (4)	C7—C8—H8A	119.5
C1—N1—Ag1	127.6 (4)	C9—C8—H8A	119.5
C2—N1—Ag1	126.6 (3)	C10—C9—C8	120.4 (5)
C1—N2—C3	107.0 (4)	C10—C9—H9A	119.8
C1—N2—C4	128.5 (4)	C8—C9—H9A	119.8
C3—N2—C4	124.4 (4)	C9—C10—C11	120.2 (5)
C5—N3—C6	117.6 (4)	C9—C10—H10A	119.9
C12—N4—C11	117.1 (4)	C11—C10—H10A	119.9
C14—N5—C15	107.3 (4)	N4—C11—C6	120.5 (5)
C14—N5—C13	125.3 (4)	N4—C11—C10	120.1 (5)
C15—N5—C13	127.4 (4)	C6—C11—C10	119.3 (4)
C14—N6—C16	106.0 (4)	N4—C12—C5	121.7 (4)
C14—N6—Ag1 ⁱ	123.6 (3)	N4—C12—C13	114.8 (4)
C16—N6—Ag1 ⁱ	130.3 (3)	C5—C12—C13	123.4 (4)
N1—C1—N2	111.2 (4)	N5—C13—C12	112.3 (3)
N1—C1—H1A	124.4	N5—C13—H13A	109.2
N2—C1—H1A	124.4	C12—C13—H13A	109.2
C3—C2—N1	110.2 (5)	N5—C13—H13B	109.2
C3—C2—H2A	124.9	C12—C13—H13B	109.2
N1—C2—H2A	124.9	H13A—C13—H13B	107.9
C2—C3—N2	106.0 (5)	N6—C14—N5	110.7 (4)
C2—C3—H3A	127.0	N6—C14—H14A	124.6
N2—C3—H3A	127.0	N5—C14—H14A	124.6
N2—C4—C5	111.8 (4)	C16—C15—N5	105.9 (5)
N2—C4—H4A	109.3	C16—C15—H15A	127.1
C5—C4—H4A	109.3	N5—C15—H15A	127.1
N2—C4—H4B	109.3	C15—C16—N6	110.1 (4)
C5—C4—H4B	109.3	C15—C16—H16A	125.0
H4A—C4—H4B	107.9	N6—C16—H16A	125.0
N3—C5—C12	121.8 (4)	F4—B1—F2	110.1 (6)
N3—C5—C4	115.6 (4)	F4—B1—F1	112.2 (7)
C12—C5—C4	122.6 (4)	F2—B1—F1	110.3 (7)
N3—C6—C11	121.2 (4)	F4—B1—F3	109.3 (7)

supplementary materials

N3—C6—C7	119.3 (5)	F2—B1—F3	111.0 (6)
C11—C6—C7	119.5 (5)	F1—B1—F3	103.8 (6)
C8—C7—C6	119.5 (5)		
Ag1 ⁱ —Ag1—N1—C1	60.4 (4)	N3—C6—C11—N4	1.3 (6)
Ag1 ⁱ —Ag1—N1—C2	-114.9 (5)	C7—C6—C11—N4	179.8 (4)
C2—N1—C1—N2	0.5 (6)	N3—C6—C11—C10	-178.8 (4)
Ag1—N1—C1—N2	-175.5 (3)	C7—C6—C11—C10	-0.3 (6)
C3—N2—C1—N1	-0.6 (6)	C9—C10—C11—N4	-179.9 (4)
C4—N2—C1—N1	176.8 (4)	C9—C10—C11—C6	0.3 (7)
C1—N1—C2—C3	-0.2 (7)	C11—N4—C12—C5	-0.1 (6)
Ag1—N1—C2—C3	175.9 (4)	C11—N4—C12—C13	-179.0 (4)
N1—C2—C3—N2	-0.2 (7)	N3—C5—C12—N4	0.4 (7)
C1—N2—C3—C2	0.5 (6)	C4—C5—C12—N4	-177.7 (4)
C4—N2—C3—C2	-177.1 (5)	N3—C5—C12—C13	179.1 (4)
C1—N2—C4—C5	8.8 (7)	C4—C5—C12—C13	1.1 (6)
C3—N2—C4—C5	-174.2 (5)	C14—N5—C13—C12	61.3 (6)
C6—N3—C5—C12	0.2 (6)	C15—N5—C13—C12	-120.5 (5)
C6—N3—C5—C4	178.4 (4)	N4—C12—C13—N5	-115.4 (5)
N2—C4—C5—N3	86.7 (5)	C5—C12—C13—N5	65.7 (6)
N2—C4—C5—C12	-95.2 (5)	C16—N6—C14—N5	-1.0 (5)
C5—N3—C6—C11	-1.1 (6)	Ag1 ⁱ —N6—C14—N5	179.5 (3)
C5—N3—C6—C7	-179.6 (4)	C15—N5—C14—N6	0.8 (5)
N3—C6—C7—C8	179.4 (4)	C13—N5—C14—N6	179.3 (4)
C11—C6—C7—C8	0.8 (7)	C14—N5—C15—C16	-0.3 (6)
C6—C7—C8—C9	-1.3 (7)	C13—N5—C15—C16	-178.8 (4)
C7—C8—C9—C10	1.2 (8)	N5—C15—C16—N6	-0.3 (6)
C8—C9—C10—C11	-0.7 (7)	C14—N6—C16—C15	0.8 (6)
C12—N4—C11—C6	-0.7 (6)	Ag1 ⁱ —N6—C16—C15	-179.7 (3)
C12—N4—C11—C10	179.5 (4)		

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

Fig. 1

