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Bis[*N*,*N*′-bis(2-fluorobenzylidene)ethylenediamine-κ^2N,N']silver(**I**) nitrate

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Key indicators

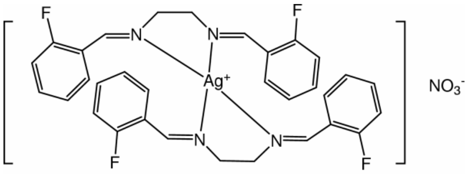
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.040
wR factor = 0.114
Data-to-parameter ratio = 15.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $[Ag(C_{16}H_{14}F_2N_2)_2]NO_3$, the Ag atom, lying on a twofold rotation axis, is four-coordinated by N atoms from two symmetry-related bidentate ethylenediamine ligands. The AgN_4 coordination forms a highly distorted tetrahedral geometry.

Comment

The propensity for d^{10} Ag^+ cations to form coordination complexes with a variety of ligands, such as those containing N, O and S donors, has been widely studied (Buchholz *et al.*, 1996; Amoroso *et al.*, 1995; Michaelides *et al.*, 1995). However, the isolation of silver(I) complexes is complicated by the fact that they often form polynuclear complexes. Moreover, from a practical viewpoint, there are serious problems, namely, nearly all polymeric silver(I) complexes are water-insoluble and most of them are unstable upon exposure to light. In this work, a new organic bis–Schiff base ligand was used to ligate with silver(I) ions, to form a water-soluble and stable complex, (I).



(I)

The title compound is an ionic complex of a bis[*N*,*N*′-bis(2-fluorobenzylidene)ethylenediamine]silver(I) cation and a nitrate anion. The asymmetric unit consists of one half of the cation and one half of the anion, with the other halves generated by a crystallographic twofold axis. Atoms Ag1 and N3 lie on the twofold axis. The Ag^I atom is four-coordinated by two N atoms from each of the two symmetry-related bidentate *N*,*N*′-bis(2-fluorobenzylidene)ethylenediamine ligands. This AgN_4 coordination forms a highly distorted tetrahedral geometry, with bond angles around the Ag atom deviating significantly from ideal values (Table 1). The Ag—N bond lengths in (I) are slightly shorter than the mean value of 2.403 Å reported by Orpen *et al.* (1989) for such distances in the coordination complexes of Schiff bases.

Experimental

At ambient temperature, an ethanol solution (5 ml) of diethylenetriamine (60 mg, 1 mmol) was added to 2-fluorobenzaldehyde (248 mg, 2 mmol) in ethanol (10 ml) with stirring. After half an hour, $AgNO_3$ (170 mg, 1 mmol) in acetonitrile (5 ml) was added to the above solution. The resultant solution was kept to evaporate slowly in air. Colorless, single crystals of the title complex, (I), were deposited and were collected by filtration. The crystals were washed with a

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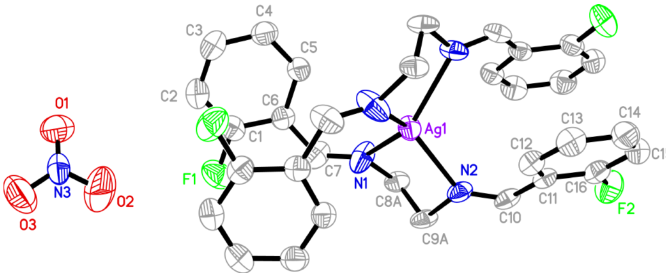

Figure 1
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Only the major components of the disordered atoms are shown and, for clarity, H atoms have been omitted.

mixture of ethanol and acetonitrile (1:1 *v*/*v*) and then dried in a vacuum desiccator over silica gel (yield 81%). Analysis calculated for (I), $C_{32}H_{26}AgF_4N_4^+\cdot NO_3^-$: C 53.95, H 3.68, N 9.83, F 10.67%; found: C 54.40, H 3.88, N 9.69, F 10.89%. IR spectrum: 3010 (*w*), 1638 (*m*), 1610 (*m*), 1485 (*m*), 1452 (*m*), 1360 (*w*), 1347 (*s*), 1301 (*m*), 1282 (*w*), 1234 (*m*), 1198 (*w*), 1193 (*w*), 1101 (*w*), 1041 (*w*), 970 (*w*), 831 (*w*), 781 (*w*), 776 (*m*), 757 (*s*).

Crystal data

$[Ag(C_{16}H_{14}F_2N_2)_2]NO_3$
M_r = 714.46
Monoclinic, $C2/c$
a = 13.0142 (15) Å
b = 18.102 (2) Å
c = 14.1367 (17) Å
β = 114.328 (2)°
V = 3034.6 (6) Å^3
Z = 4

D_x = 1.564 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4783 reflections
θ = 2.8–28.3°
μ = 0.73 mm^{-1}
T = 293 (2) K
Block, light yellow
0.46 × 0.36 × 0.32 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.730, $T_{\max}$ = 0.800
9317 measured reflections

3699 independent reflections
2832 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.022
$\theta_{\max}$ = 28.3°
$h = -17 \rightarrow 13$
$k = -23 \rightarrow 17$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.114
S = 1.09
3699 reflections
237 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 2.0621P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.71 e Å^{-3}
$\Delta\rho_{\min}$ = −0.46 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—N1	2.280 (3)	N1—C7	1.256 (4)
Ag1—N2	2.334 (2)	N2—C10	1.255 (4)
N1$^{\rm i}$—Ag1—N1	122.7 (2)	N1—Ag1—N2	76.24 (9)
N1$^{\rm i}$—Ag1—N2	138.38 (12)	N2$^{\rm i}$—Ag1—N2	115.74 (13)

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

Atoms C8 and C9 of the ethylenediamine group were found to be disordered. The occupancies of the disordered positions C8*A*/C8*B* and C9*A*/C9*B* were refined to 0.55 (1)/0.45 (1). The corresponding N—C and C—C distances in the major and minor conformers were restrained to be equal. The N atom of the NO_3^- anion lies on a twofold axis and hence there are two positions for each O atom, related by the symmetry operation ($1 - x$, y, $\frac{1}{2} - z$), with equal occupancies. Significant deviations in the N—O bond lengths were observed and attempts to restrain them resulted in an unstable refinement. The H atoms were geometrically positioned and were treated as riding atoms on the parent C atoms, with C—H distances of 0.93 or 0.97 Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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