Table 2. *Hydrogen-bonding geometry (Å, °)*

| D—H⋯A | D—H | H⋯A | D⋯A | D—H⋯A |
|---|---|---|---|---|
| N3—H01⋯O10 | 0.839 (16) | 2.36 (3) | 3.083 (4) | 144 (3) |
| N5—H05⋯O9ⁱ | 0.839 (16) | 2.20 (2) | 2.971 (4) | 153 (3) |
| N8—H012⋯O12 | 0.833 (16) | 2.159 (19) | 2.963 (4) | 162 (4) |
| N10—H016⋯O11ⁱⁱ | 0.842 (16) | 2.49 (3) | 3.062 (4) | 126 (3) |
| N3—H01⋯O1ⁱⁱⁱ | 0.839 (16) | 2.50 (3) | 2.957 (4) | 115 (3) |
| N3—H02⋯O6ⁱᵛ | 0.840 (16) | 2.19 (2) | 2.980 (4) | 157 (3) |
| N4—H03⋯O8ⁱᵛ | 0.831 (16) | 2.118 (19) | 2.924 (4) | 163 (3) |
| N4—H04⋯O4ᵛ | 0.828 (15) | 2.186 (18) | 2.992 (4) | 165 (3) |
| N6—H07⋯O3 | 0.850 (15) | 2.25 (2) | 2.996 (4) | 146 (3) |
| N6—H08⋯O1ⁱⁱⁱ | 0.831 (15) | 2.220 (17) | 3.047 (4) | 173 (3) |
| N7—H09⋯O7 | 0.836 (15) | 2.066 (19) | 2.881 (4) | 165 (4) |
| N7—H010⋯O4 | 0.840 (16) | 2.23 (3) | 2.970 (4) | 147 (4) |
| N8—H011⋯O2 | 0.838 (15) | 2.109 (19) | 2.909 (4) | 160 (3) |
| N8—H011⋯O5ᵛⁱ | 0.838 (15) | 2.60 (3) | 2.864 (4) | 100 (3) |
| N8—H012⋯O5ᵛⁱ | 0.833 (16) | 2.60 (4) | 2.864 (4) | 100 (3) |
| N9—H013⋯N99 | 0.836 (16) | 2.22 (2) | 3.024 (5) | 160 (4) |
| N9—H014⋯O5ᵛⁱ | 0.834 (16) | 2.123 (18) | 2.944 (4) | 168 (4) |
| N10—H015⋯N99 | 0.851 (15) | 2.63 (3) | 3.328 (6) | 140 (3) |
| N10—H016⋯O7ⁱⁱ | 0.842 (16) | 2.34 (3) | 2.970 (4) | 132 (3) |

Symmetry codes: (i) $1 - x, -1 - y, 1 - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$; (iii) $1 - x, y, \frac{3}{2} - z$; (iv) $1 - x, 1 - y, 1 - z$; (v) $1 - x, -y, 1 - z$; (vi) $x, 1 - y, \frac{1}{2} + z$.

NH were freely refined but with N—H bond lengths restrained as equal (command *SADI*; Sheldrick, 1997); methyl-H atoms were located from difference syntheses, idealized and refined as rigid groups allowed to rotate, but not tip.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *XP* (Siemens, 1994b). Software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1351). Services for accessing these data are described at the back of the journal.

## References

Blaschette, A., Hippel, I., Krahl, J., Wieland, E., Jones, P. G. & Sebald, A. (1992). *J. Organomet. Chem.* **437**, 279–297.

Fait, J. (1991). *XSCANS User's Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Henschel, D., Moers, O., Blaschette, A. & Jones, P. G. (1997). *Acta Cryst.* C53, 1877–1879.

Hiemisch, O., Henschel, D., Blaschette, A. & Jones, P. G. (1999). *Z. Anorg. Allg. Chem.* **625**, 1391–1394.

Hippel, I., Jones, P. G. & Blaschette, A. (1993). *J. Organomet. Chem.* **448**, 63–67.

Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.

Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Siemens (1994a). *XEMP. Empirical Absorption Correction Program.* Version 2.10. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1994b). *XP. Molecular Graphics Program.* Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Tavridou, P., Russo, U., Marton, D., Valle, G. & Kovala-Demertzi, D. (1995). *Inorg. Chim. Acta*, **231**, 139–145.

Tavridou, P., Russo, U., Valle, G. & Kovala-Demertzi, D. (1993). *J. Organomet. Chem.* **460**, C16–18.

Weitze, A., Henschel, D., Blaschette, A. & Jones, P. G. (1995). *Z. Anorg. Allg. Chem.* **621**, 1746–1754.

Wirth, A. (1998). PhD thesis, Technische Universität Braunschweig, Germany. [ISBN 3-89720-163-1]

Wirth, A., Blaschette, A. & Jones, P. G. (1998). *Main Group Met. Chem.* **21**, 309–313.

Wirth, A., Moers, O., Blaschette, A. & Jones, P. G. (1998). *Z. Anorg. Allg. Chem.* **624**, 1686–1694.

Wirth, A., Moers, O., Blaschette, A. & Jones, P. G. (1999). *Z. Anorg. Allg. Chem.* **625**, 982–988.

# [2-(2-Pyridyl-N)quinoxaline-N¹]silver(I) nitrate

S. Shanmuga Sundara Raj,[a] Hoong-Kun Fun,[a] Xiao-Feng Chen,[b] Xu-Hui Zhu[b] and Xiao-Zeng You[b]

[a]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and* [b]*Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China. E-mail: hkfun@usm.my*
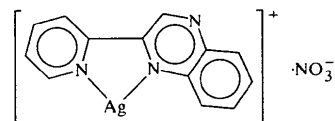
## Abstract

The title compound, [Ag(C₁₃H₉N₃)]NO₃, is an infinite one-dimensional polymer. Each Ag atom is coordinated by three N atoms, with Ag—N bond distances in the range of 2.246 (3)–2.413 (3) Å.

## Comment

Much effort has been devoted recently to the design of metal complexes exhibiting helical or polymeric architectures which have particular relevance to biological systems and asymmetric catalysis. Several silver(I) polymer helices have been reported (Tong *et al.*, 1998; Hester *et al.*, 1997; Carlucci *et al.*, 1998; Psillakis *et al.*, 1997). In this paper, we report the crystal structure of a silver(I)–2-(2-pyridyl)quinoxaline complex, (I).



(I)

Each Ag atom is, as expected in a three-coordinate environment, linked to three N atoms from two different 2-(2-pyridyl)quinoxaline molecules, resulting in trigonal coordination distorted toward Y-shaped geometry [N3—Ag1—N2($x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$) 141.2 (1)°, N1—
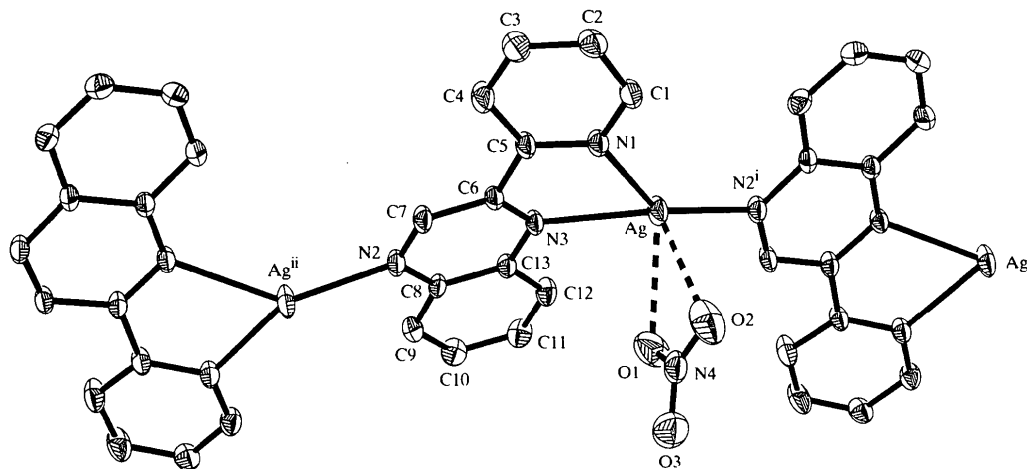
Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry codes: (i) $x - \frac{1}{2}$, $\frac{1}{2} - y$, $z - \frac{1}{2}$; (ii) $\frac{1}{2} + x$, $\frac{1}{2} - y$, $\frac{1}{2} + z$.]

Ag1—N3 70.9 (1)° and N1—Ag1—N2($x - \frac{1}{2}$, $-y + \frac{1}{2}$, $z - \frac{1}{2}$) 139.2 (1)°]. The Ag—N bond distances lie in the range of 2.246 (3)–2.413 (3) Å, comparable to the range of 2.211–2.520 Å found in Ag{bis[3-(2-pyridyl)-pyrazol-1-yl]phosphinate}·2H₂O (Psillakis *et al.*, 1997). Successive Ag atoms and quinoxaline molecules are glide-plane related, resulting in infinite polymeric chains parallel to the *ac* diagonal.

There are weak interactions between the Ag¹ atom and a nitrate ion. The Ag1···O1 and Ag1···O2 distances are 2.684 (5) and 2.739 (7) Å, respectively, rather longer than a typical interaction between Ag¹ and nitrate (2.093 Å; Tong *et al.*, 1998). The nitrate ions are also linked by C—H···O interactions to neighbouring Ag–quinoxaline chains. Within each ligand molecule, the pyridyl and quinoxalinyl groups make an interplanar angle of 18.9 (2)°.

## Experimental

The title compound was prepared by adding a methanolic solution of 2-(2-pyridyl)quinoxaline (Kasselouri *et al.*, 1993) to a stirred methanolic solution of AgNO₃ at 323 K to give a clear mixture. After several days, yellow single crystals were obtained by slow evaporation at room temperature.

*Crystal data*

[Ag(C₁₃H₉N₃)]NO₃
$M_r = 377.11$
Monoclinic
$Cc$
$a = 14.1570\,(2)$ Å
$b = 12.9188\,(2)$ Å
$c = 7.2671\,(1)$ Å
$\beta = 104.776\,(1)°$
$V = 1285.14\,(3)$ Å³
$Z = 4$
$D_x = 1.949$ Mg m⁻³
$D_m$ not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 3747 reflections
$\theta = 2.17–28.32°$
$\mu = 1.585$ mm⁻¹
$T = 293\,(2)$ K
Parallelepiped
$0.32 \times 0.18 \times 0.10$ mm
Yellow

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.631$, $T_{max} = 0.858$
4401 measured reflections
1594 independent reflections (plus 1243 Friedel-related reflections)

2490 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 28.29°$
$h = -18 \rightarrow 18$
$k = -13 \rightarrow 17$
$l = -9 \rightarrow 9$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.078$
$S = 0.968$
2837 reflections
190 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.691$ e Å⁻³
$\Delta\rho_{min} = -0.243$ e Å⁻³
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure:
  Flack (1983)
Flack parameter = 0.02 (3)

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Ag1—N1 | 2.315 (3) | N1—C5 | 1.348 (5) |
| Ag1—N3 | 2.413 (3) | N2—C7 | 1.299 (5) |
| O1—N4 | 1.232 (7) | N2—Ag1ⁱ | 2.246 (3) |
| O2—N4 | 1.226 (6) | N3—C6 | 1.314 (5) |
| O3—N4 | 1.226 (7) | N3—C13 | 1.357 (5) |
| N1—C1 | 1.329 (5) | | |
| N1—Ag1—N3 | 70.9 (1) | C6—N3—Ag1 | 113.8 (3) |
| C1—N1—Ag1 | 123.4 (3) | C13—N3—Ag1 | 125.0 (2) |
| C5—N1—Ag1 | 117.7 (2) | | |

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

Table 2. *Hydrogen-bonding geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C3—H3A···O2ⁱ | 0.93 | 2.54 | 3.434 (7) | 162 |
| C10—H10A···O2ⁱⁱ | 0.93 | 2.50 | 3.227 (7) | 136 |
| C11—H11A···O1ⁱⁱⁱ | 0.93 | 2.56 | 3.459 (7) | 164 |

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

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* and *PARST* (Nardelli, 1995).

## References

Carlucci, L., Ciani, G., Proserpio, D. M. & Sironi, A. (1998). *Inorg. Chem.* **37**, 5941–5943.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Hester, C. A., Baughman, R. G. & Collier, H. L. (1997). *Polyhedron*, **16**, 2893–2895.

Kasselouri, S., Garoufis, A., Katehanakis, A., Kalkanis, G., Perlepos, S. P. & Hadjiliadis, N. (1993). *Inorg. Chim. Acta*, **207**, 255–258.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Psillakis, E., Jeffery, J. C., McCleverty, J. A. & Ward, M. D. (1997). *J. Chem. Soc. Dalton Trans.* pp. 1645–1651.

Sheldrick, G. M. (1996). *SADABS. Program for Empirical Absorption Correction of Area Detector Data.* University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXTL Software Reference Manual.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Tong, M. L., Chen, X. M., Ye, B. H. & Ng, S. W. (1998). *Inorg. Chem.* **37**, 5278–5281.

# Dioxobis(8-quinolinolato-*N,O*)tungsten(VI)

S. Shanmuga Sundara Raj,[a] Hoong-Kun Fun,[a] Zhen-Feng Chen,[b] Jing Zhang,[b] Xiang Xue,[b] Ren-Gen Xiong[b] and Xiao-Zeng You[b]

[a]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and* [b]*Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China. E-mail: hkfun@usm.my*
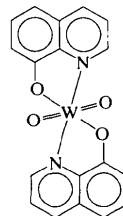
## Abstract

The crystal structure of the title complex, [WO$_2$(C$_9$H$_6$-NO)$_2$], a hexacoordinated molecule, has distorted octa-hedral geometry with $C_2$ site symmetry. There are two terminal O ligands with a short W—O bond length [1.722 (4) Å] and a large O—W—O angle [106.6 (3)°] in the molecule. The two terminal O ligands are in *cis* positions. The molecules join to form a two-dimensional network parallel to (001) through C—H···O hydrogen bonds.

## Comment

8-Quinolinol has been used widely to determine the quantity of tungsten and molybdenum (Marcel & Rene, 1950). The complexes of 8-quinolinol with niobium, molybdenum, tungsten and hafnium emit interesting fluorescence (Schneider & Roselli, 1970). Photocatalyzed synthesis, characterization and thermal behavior of tungsten(IV) complexes with oxine has been studied (Ali & Yunhir, 1993). Although dioxotungsten complexes with oxine were prepared by various methods, so far, the hydrothermal treatment of carbonyl tungsten, 8-quinolinol and methanol has not been reported. Moreover, the crystal structure of the title compound, (I), to the best of our knowledge, has not been reported previously.



(I)

The bond angles in the aromatic ring system are found to be between 118.2 (5) and 121.7 (5)°. In the 8-quinolinolate ligands, the absence of any unusually long bonds and the marginally longer carbonyl bond [1.355 (6) Å *versus* 1.28 Å], shorter than the normal single bond in ether and alcohols (>1.4 Å), suggest that the delocalization extends over the entire molecule and will therefore withdraw more negative charge from the cation (Barton *et al.*, 1983).

The two bidentate ligands are arranged at an angle of 83.2 (1)° with respect to one another in a *cis* geometry, and the W atom therefore represents a center of chirality. The *cis* topology is in agreement with several examples of hexacoordinate complexes with bidentate ligands like *o*-phenanthroline, bipyridine or 8-quinoline (Sawitzki *et al.*, 1978; Bellavance *et al.*, 1977; Klebe & Tran Qui, 1984), and can be explained in stereochemical terms: the *cis* geometry of the ligands minimizes the steric interference between the H atoms in the 2 and 4 positions of the different ring systems. In the title complex, the chelate ligands are connected to the central W atom through a W—O single bond and a W—N coordinative bond. The two W—O (O2) bonds form