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ARTICLE TYPE

Pyridine N-Oxide: a Hyperdentate Argentophile

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5 Pyridine N-oxide reacts with silver(I) perchlorate monohydrate in three different stoichiometric ratios to produce three very different complexes all of which are 1-D coordination polymers. Reaction with silver(I) triflate also furnishes a crystalline 1-D polymer. In three of the four complexes the pyridine N-oxide acts as a rare μ_3 -O,O,O triply bridging ligand, for which we propose the term “hyperdentate”.

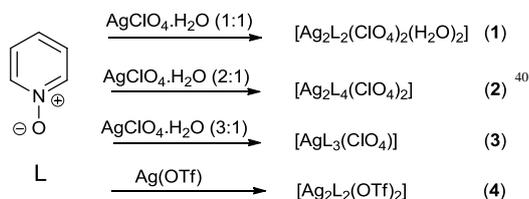
Introduction

10 Metallosupramolecular chemistry involves the use of a combination of bridging organic ligands with metal centres to generate 0-, 1-, 2- or 3-D assemblies.¹ Linear bridging N-heterocyclic ligands, such as pyrazine and 4,4'-bipyridine, have been much used to prepare numerous coordination polymers of varying dimensionality.² Silver(I) has recently proved popular
15 as a metal in metallosupramolecular synthesis because of its promiscuous coordination ability.³ The chemistry of silver with pyrazine derivatives has recently been reviewed.⁴ We have now embarked on a study of the reactions of silver(I) with the N-oxides of bridging N-heterocyclic ligands, but before doing so we decided to explore the coordination chemistry of silver with
20 pyridine N-oxide itself.

Pyridine N-oxide plays a special role in organic chemistry since, unlike pyridine, it readily undergoes electrophilic substitution reactions.⁵ It is also often used as a ligand in coordination chemistry but, curiously, there is only one report⁶ of a silver complex of pyridine N-oxide in the Cambridge
25 Structural Database.⁷ We now report that pyridine N-oxide has a special attraction for silver(I), being able to bond to one, two or three silver atoms, depending on the reaction stoichiometry and the nature of the counterion.

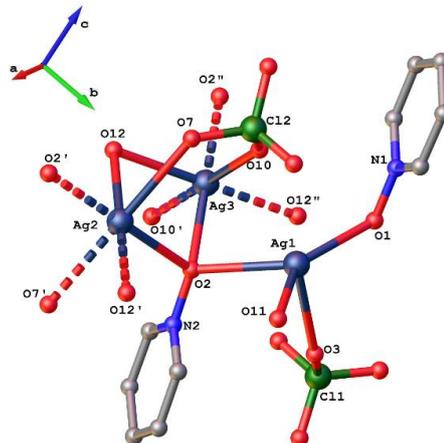
Results and discussion

30 Pyridine N-oxide (L) was reacted with silver(I) perchlorate monohydrate in three different stoichiometric ratios to produce three different crystalline products (Scheme 1). Reaction in a 1:1 ratio consistently furnished a product (1) that crystallises in the triclinic space group P-1. The asymmetric unit is shown in Fig. 1 with selected atom labels and bonding parameters and comprises three independent silver(I) ions, two of which lie on
35 crystallographic inversion centres, two ligands L, two perchlorate anions and two water molecules. Of the two independent N-oxides one is monodentate while the other is μ_3 -O,O,O tridentate.



45 **Scheme 1** Synthesis of complexes.

One silver atom (Ag1) is four-coordinate, being bound to two N-oxide oxygens, a monodentate perchlorate and a water molecule and has a seesaw
50 geometry [$\tau_4 = 0.67$].⁸ The other two silver atoms are six-coordinate with distorted octahedral geometries and are each bound to two oxygens from bridging N-oxides, two oxygens from bridging perchlorates and two bridging water oxygens. Surprisingly, the N-O bond lengths are the same for the monodentate and μ_3 -bridging L ligands. However, the Ag-O bond
55 lengths are considerably longer to the bridging ligand than to the monodentate one, as might be expected on steric grounds. The μ_3 -O,O,O tridentate oxygen (O2) has a distorted tetrahedral geometry with angles ranging from 92.84(4)° to 122.94(9)°.



60 **Fig. 1** The asymmetric unit of complex 1. Hydrogen atoms are excluded for clarity. Symmetry related atoms in the adjacent asymmetric unit are shown with dashed bonds. Selected bond lengths (Å) and bond angles (°): N1-O1 = 1.347(2), N2-O2 = 1.349(2), Ag1-O1 = 2.335(1), Ag1-O2 = 2.455(1), Ag2-O2 = 2.413(1), Ag3-O2 = 2.511(1), N1-O1-Ag1 = 115.65(9), N2-O2-Ag2 = 112.84(9), N2-O2-Ag1 = 115.19(9), N2-O2-Ag3 = 122.94(9), Ag2-O2-Ag1 = 113.39(5), Ag2-O2-Ag3 = 97.28(4), Ag1-O2-Ag3 = 92.84(4), O-Ag1-O range 82.89(4) – 142.39(5), O-Ag2-O range 80.23(4) – 180, O-Ag3-O range 75.59(5) – 180.

70 There are many pyridine N-oxide metal complexes in the Cambridge Structural Database. Of these thirty six involve the oxygen bridging two metal centres (mainly copper, manganese and cadmium). Interestingly, the only example of pyridine N-oxide bridging three metal centres also involves silver, this being in a structure where it decorates the surface of an Ag_{14} cluster.⁶ Formally this is possible if the oxygen is considered to be sp^3
75 hybridised with three lone pairs available as donors. We propose the term “hyperdentate” to describe a situation where an atom binds to more metals than normal. For example, the archetypical ligand pyridine normally only

binds to one metal centre but there are three reported structures of the nitrogen being hyperdentate and bridging two metals,⁹ one of which coincidentally also involves silver.^{9a}

The structure of **1** extends into a 1-D coordination polymer that propagates along the *a* axis. As shown in Fig. 2 the tetrahedron formed by the μ_3 -L and the three silver atoms connect in a vertex-sharing fashion through the octahedral silver centres. Adjacent polymer chains are further interconnected by hydrogen bonding involving the coordinated water molecules and the monodentate N-oxide oxygen.

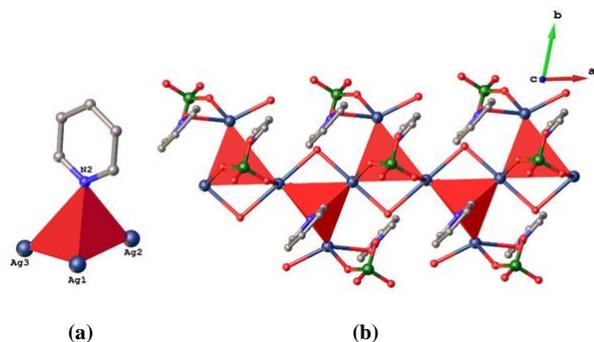


Fig. 2 (a) The tetrahedron formed by coordination of the tridentate pyridine N-oxide with three different silvers. (b) Vertex-sharing representation of the 1-D polymeric structure of complex **1** viewed down the *c* axis.

Reaction of **L** with silver perchlorate monohydrate in a 2:1 ligand to metal ratio consistently produced a product (**2**) in up to 84% yield. This crystallises in the trigonal space group *R*3, with the asymmetric unit containing four molecules of **L**, two perchlorates (one of which is coordinated) and four independent silver atoms, three of which lie on a three-fold rotation axis (Fig. 3).

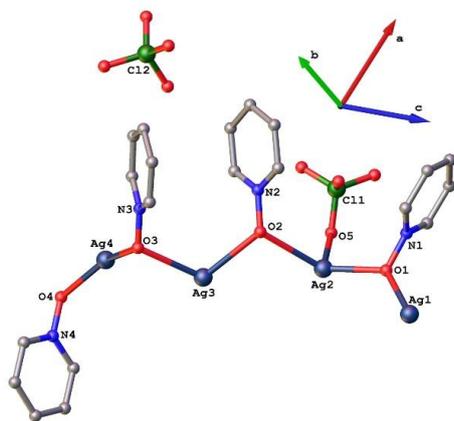


Fig. 3 The asymmetric unit of complex **2**. Hydrogen atoms are excluded for clarity. Selected bond lengths (Å): Ag1-O1 = 2.471(2), Ag2-O1 = 2.192(2), Ag2-O2 = 2.275(2), Ag2-O5 = 2.521(3), Ag3-O2 = 2.515(2), Ag3-O3 = 2.475(2), Ag4-O3 = 2.490(2), Ag4-O4 = 2.464(2).

The structure extends into a 1-D coordination polymer (Fig. 4) which propagates along the *c* axis and consists of repeating units of three silver atoms lying on the three-fold rotation axis separated by Ag_3 triangles generated from the full occupancy (Ag_2) silver. Of the four independent **L** molecules three have μ_2 -coordination and the other has hyperdentate μ_3 -coordination. These four rings spiral around the chains of silver atoms in a manner that allows for π - π interactions, with centroid-centroid distances of 3.57, 3.60 Å and 3.76 Å between the rings.

All silver atoms are six-coordinate with highly distorted octahedral geometries, with those lying on the three-fold axis being bound to six different pyridine N-oxide oxygens. The silver that forms part of the triangular array coordinates to three N-oxide oxygens, a perchlorate oxygen and two symmetry related silvers. The Ag-Ag bond distance in the triangle is 3.019(4) Å. On average the Ag-O bond lengths are shorter to the doubly bridging N-oxides than to the hyperdentate triply bridging one.

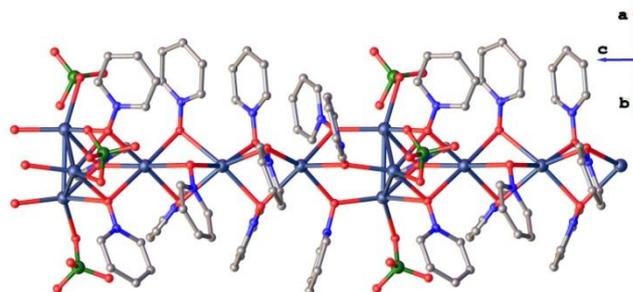


Fig. 4 The 1-D polymeric assembly of complex **2**. Hydrogen atoms and non-coordinated perchlorate anions are omitted for clarity.

An analogous reaction in a 3:1 ligand to metal ratio produced yet another product (**3**) in 38% yield. It crystallises in the hexagonal space group *P*6₃/*m* with the asymmetric unit comprising an **L** molecule lying on a mirror plane, a silver atom and a disordered perchlorate anion each of which lie on three-fold axes. The overall stoichiometry is 1:3 as used in the reaction itself. The pyridine N-oxide oxygen bridges two symmetry related silver atoms which are bonded to one another with a separation of 2.996(1) Å. The silver atom is eight-coordinate being bonded to six symmetry related **L** oxygens and two adjacent silvers.

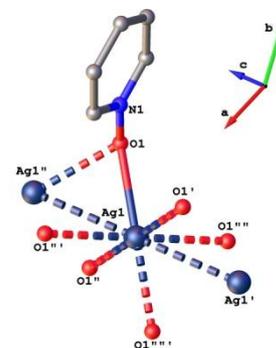


Fig. 5 Asymmetric unit of complex **3**. The disordered perchlorate and hydrogen atoms are omitted for clarity. Symmetry related atoms are shown with dashed bonds. Selected bond lengths (Å) and bond angles (°): N1-O1 = 1.328(6), Ag1-O1 = 2.482(3), Ag1-Ag1' = 2.9959(1), N1-O1-Ag1 = 117.4(2), Ag1-O1-Ag1'' = 74.3(1), Ag1'-Ag1-Ag1' = 180.0.

The structure extends into a 1-D coordination polymer (Fig. 6) that propagates along the *c* axis and consists of linear chains of bonded silver atoms, a “silver wire”,¹⁰ with adjacent atoms bridged by three molecules of pyridine N-oxide. The perchlorate anions fill the spaces between the linear chains.

Thus, three different silver perchlorate complexes have been prepared simply by changing the stoichiometry. Molar-ratio-dependent supramolecular isomerism of silver coordination polymers has recently been reported by another group.¹¹ It is noteworthy that as the ratio of ligand increases the proportion of hyperdentate μ_3 -*O,O,O* oxygens decreases from 50% in **1** to 33% in **2** to 0% in **3**, suggesting that hyperdentate is favoured by a high ratio of silver. Accordingly, we examined reactions with 1:2

1:3 L:M ratios but this simply reproduced complex **1**. Similarly a 6:1 ratio produced complex **3**. We also explored other variables such as the solvent. Reaction in a 1:1 ratio in a variety of solvent combinations invariably led to the same complex **1**. Thus, we next examined the effect of the counterion.

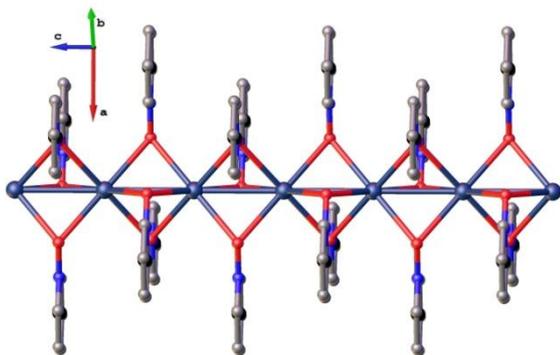


Fig. 6 1-D polymeric structure of complex **3**.

Reaction of L with silver(I) triflate in a 1:1 ratio gave a different complex (**4**), which crystallises in the triclinic space group P-1. The asymmetric unit (Fig. 7) contains two independent silver atoms, two L molecules and two triflate anions. The N-oxide and triflate ligands each display both μ_2 - and μ_3 -bridging modes of coordination. In this case the average Ag-O bond length to the hyperdenate μ_3 -oxygen [2.411(2) Å] is only slightly longer than to the μ_2 -oxygen [2.395(2) Å]. There is one weak Ag-Ag interaction [3.247(3) Å] between symmetry related Ag2 atoms; Ag1 and Ag2 are separated by 3.381(4) Å. Ignoring the weak Ag-Ag interaction, the two silvers are five coordinate with τ_5 values¹² of 0.38 for Ag1 and 0.21 for Ag2, indicating geometries closer to square pyramidal than trigonal bipyramidal.

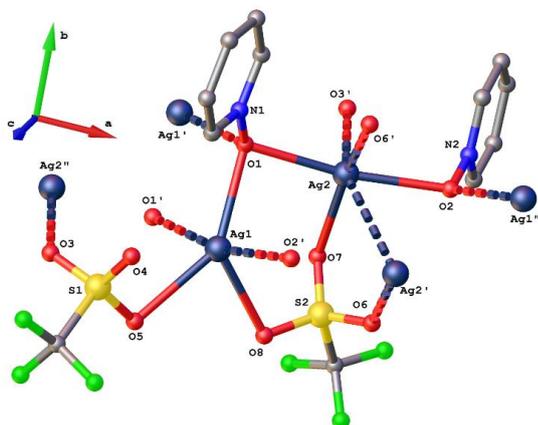


Fig. 7 Asymmetric unit of complex **4**. Hydrogens atoms are omitted for clarity. Selected bond lengths (Å): N1-O1 = 1.349(2), N2-O2 = 1.336(2), Ag1-O1 = 2.417(2), Ag1-O1' = 2.390(2), Ag1-O2' = 2.371(2), Ag1-O5 = 2.528(2), Ag1-O8 = 2.464(2), Ag2-O1 = 2.418(2), Ag2-O2 = 2.380(2), Ag2-O7 = 2.479(2), Ag2-O6' = 2.456(2), Ag2-O3' = 2.479(2), Ag2-Ag2' = 3.2472(3), N1-O1-Ag1' = 112.19(11), N1-O1-Ag1 = 118.95(12), N1-O1-Ag2 = 116.41(12), N2-O2-Ag1'' = 113.14(13), N2-O2-Ag2 = 110.07(12), Ag1''-O2-Ag2 = 124.44(7), Ag1'-O1-Ag2 = 117.44(6).

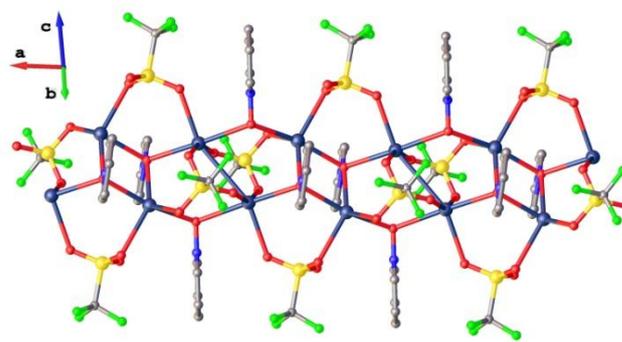
Once again the complex is a 1-D coordination polymer that this time propagates along the *a* axis (Fig. 8). The ladder-like chains of silver atoms are held together by bridging N-oxide ligands and triflate anions.

Conclusions

In conclusion, we have shown that pyridine N-oxide has a special affinity for silver(I) and that it is possible to prepare very different metallosupramolecular assemblies simply by varying the stoichiometric ratio of the reactants. In three of the four structures described the pyridine

N-oxide oxygen acts in a μ_3 -hyperdentate mode, which has previously been reported only once before.

Fig. 8 1-D polymeric structure of complex **4**.



Experimental Section

General

Unless otherwise specified, all reagents and starting materials were reagent grade, purchased from standard suppliers and used as received. Melting points were recorded on an electrothermal melting point apparatus and are uncorrected. Elemental analysis was carried out by Campbell Microanalytical Laboratory, University of Otago. Infrared spectra were recorded on a Perkin-Elmer Spectrum One FTIR instrument operating in diffuse reflectance mode with samples prepared as KBr mulls (KBr). Mass spectra were recorded on either a DIONEX Ultimate 3000 or Bruker MaXis 4G spectrometer, both of which were operated in high resolution positive ion electrospray mode. Samples were dissolved and diluted to the required concentration in HPLC grade methanol.

Preparation of complexes

Complex (1)

Silver(I) perchlorate monohydrate (32 mg, 0.15 mmol) dissolved in methanol (2 ml) was added to an acetone (2 ml) solution of ligand L (15 mg, 0.15 mmol). The solution was left in darkness at room temperature and allowed to evaporate slowly to give colourless crystals suitable for X-ray crystallography. Yield 24 mg (53%), m.p. 78°C. Elemental Analysis: calculated for $C_5H_5NO \cdot AgClO_4$: C, 19.86; H, 1.67; N, 4.63. Found: C, 19.43; H, 2.00; N, 4.32. IR (KBr) ν_{max}/cm^{-1} : 3103, 2012, 1649, 1469, 1505, 1232, 1140, 1090, 837, 770, 675. ESI-MS m/z : Found $[L+Ag]^+$ 201.9414, C_5H_5AgNO requires $[L+Ag]^+$ 201.9417.

Complex (2)

A solution of silver(I) perchlorate monohydrate (10 mg, 0.048 mmol) in methanol (1 ml) was added to a solution of ligand L (9.1 mg, 0.096 mmol) in methanol (2 ml). The mixture was then left in darkness and allowed to evaporate slowly at room temperature, with occasional agitation from earthquakes and aftershocks. This produced colourless crystals suitable for X-ray crystallography. Yield 16 mg (84%), m.p. 88°C. IR (KBr) ν_{max}/cm^{-1} : 3110, 1653, 1466, 1226, 1176, 1144, 1086, 1019, 835, 770, 674. ESI-MS m/z : Found $[L+Ag]^+$ 201.9415, C_5H_5AgNO requires $[L+Ag]^+$ 201.9417; Found $[2L+Ag]^+$ 296.9785, $C_{10}H_{10}AgN_2O_2$ requires $[2L+Ag]^+$ 296.9788.

Complex (3)

Silver(I) perchlorate monohydrate (10 mg, 0.048 mmol) dissolved in methanol (1 ml) was added to a methanol (2 ml) solution of ligand L (13.7 mg, 0.144 mmol). The solution was left in darkness at room temperature and allowed to evaporate slowly to give crystals suitable for X-ray analysis. Yield 9 mg (38%), m.p. 67°C. Elemental Analysis: calculated for $3(C_5H_5NO) \cdot AgClO_4$: C, 36.57; H, 3.06; N 8.53. Found: C, 37.06; H, 3.14;

N, 8.60. IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3110, 1659, 1469, 1226, 1177, 1120, 1140, 1019, 835, 771, 675. ESI-MS m/z : No $[\text{L}+\text{Ag}]^+$ ions observed.

Complex (4)

A methanol (1ml) solution of silver(I) triflate (37.8 mg, 0.147 mmol) was added into a methanol (3ml) solution of ligand L (14 mg, 0.14 mmol). The solution was left in darkness at room temperature and allowed to evaporate slowly to give crystals suitable for X-ray analysis. Yield 14 mg (27%), m.p. 122°C. Elemental Analysis: calculated for $\text{C}_5\text{H}_5\text{NO}\cdot\text{AgSO}_3\text{CF}_3$: C, 20.47; H, 1.43; N, 3.98. Found: C, 20.73; H, 1.51; N, 3.97. IR (KBr) $\nu_{\text{max}}/\text{cm}^{-1}$: 3121, 2465, 1686, 1473, 1266, 1166, 1037, 838, 766, 673, 646. ESI-MS m/z : Found $[\text{L}+\text{Ag}]^+$ 201.9416, $\text{C}_5\text{H}_5\text{AgNO}$ requires $[\text{L}+\text{Ag}]^+$ 201.9417.

Crystallography

X-Ray crystallographic data collection was carried out with a Bruker APEXII or Agilent Supernova instrument, using Mo $K\alpha$ ($\lambda = 0.71073 \text{ \AA}$) radiation. All structures were solved using direct methods with SHELXS and refined on F2 using all data by full matrix least-squares procedures with SHELXL.¹³ Unless otherwise stated all non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were included in calculated positions with isotropic displacement parameters 1.2 times the isotropic equivalent of their carrier atoms. Experimental details are listed in Table 1.

Acknowledgements

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Notes and references

Electronic Supplementary Information (ESI) available: X-ray crystallographic data as CIFs for compounds **1** - **4**. CCDC numbers: 965104 (**1**), 965105 (**2**), 965106 (**3**), and 965107 (**4**). See DOI: 10.1039/b000000x/

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ARTICLE TYPE

Table 1. X-Ray crystallography experimental details

| Complex | (1) | (2) | (3) | (4) |
|---|--|--|--|---|
| Empirical formula | C ₁₀ H ₁₄ N ₂ O ₁₂ Cl ₂ Ag ₂ | C ₂₀ H ₂₀ N ₄ O ₁₂ Cl ₂ Ag ₂ | C ₁₅ H ₁₅ N ₃ O ₈ ClAg | C ₁₂ H ₁₀ N ₂ O ₈ S ₂ F ₆ Ag ₂ |
| Formula weight | 640.87 | 795.04 | 508.62 | 704.08 |
| Temperature K | 113.0 | 120.0 | 120.0 | 120.0 |
| Crystal system | Triclinic | Trigonal | Hexagonal | Triclinic |
| Space group | <i>P</i> -1 | <i>R</i> 3 | <i>P</i> 6 ₃ / <i>m</i> | <i>P</i> -1 |
| Unit cell dimensions: <i>a</i> /Å | 7.3920(2) | 21.9957(2) | 13.3014(3) | 8.26718(8) |
| <i>b</i> /Å | 10.7920(3) | 21.9957(2) | 13.3014(3) | 10.26639(13) |
| <i>c</i> /Å | 11.6410(4) | 14.0739(1) | 5.9918(2) | 11.74637(13) |
| <i>α</i> /° | 80.286(1) | 90.00 | 90.00 | 86.5046(9) |
| <i>β</i> /° | 88.272(1) | 90.00 | 90.00 | 81.1991(9) |
| <i>γ</i> /° | 77.080(1) | 120.00 | 120.00 | 88.2690(9) |
| Volume/Å ³ | 892.14(5) | 5896.88(10) | 918.09(5) | 983.172(19) |
| Z | 2 | 9 | 2 | 2 |
| Density (calculated) Mg/m ³ | 2.386 | 2.015 | 1.840 | 2.378 |
| Absorption coefficient mm ⁻¹ | 2.562 | 1.768 | 1.294 | 2.306 |
| F(000) | 624 | 3528 | 508 | 680 |
| Crystal size/mm ³ | 0.48 x 0.37 x 0.13 | 0.31 x 0.27 x 0.13 | 0.22 x 0.14 x 0.10 | 0.20 x 0.16 x 0.09 |
| Theta range for data collection (°) | 4.84 to 60.00 | 6.18 to 54.94 | 6.12 to 54.86 | 5.46 to 55.00 |
| Reflections collected [R(int)] | 24007 [0.0322] | 95119 [0.0573] | 22378 [0.0376] | 148021 [0.0528] |
| Independent reflections | 5214 | 6016 | 767 | 4516 |
| Completeness % | 99.0 | 99.9 | 99.9 | 99.9 |
| Data/restraints/parameters | 5214/0/272 | 6016/1/361 | 767/0/56 | 4520/0/289 |
| Goodness-of-fit on F ² | 1.023 | 1.159 | 1.169 | 1.095 |
| Final R ₁ indices [I>2σ(I)] | R ₁ = 0.0211 wR ₂ = 0.0547 | R ₁ = 0.0203 wR ₂ = 0.0504 | R ₁ = 0.0412 wR ₂ = 0.1014 | R ₁ = 0.0206 wR ₂ = 0.0476 |
| Final R indices [all data] | R ₁ = 0.0220 wR ₂ = 0.0553 | R ₁ = 0.0215 wR ₂ = 0.0512 | R ₁ = 0.0454 wR ₂ = 0.1047 | R ₁ = 0.0239 wR ₂ = 0.0500 |
| Largest diff. peak/hole / e Å ⁻³ | 0.53/-0.92 | 0.76/-0.42 | 0.87/-1.00 | 0.72/-0.54 |

Pyridine N-Oxide: a Hyperdentate Argentophile

Rakesh Puttreddy and Peter J. Steel

Graphical Abstract

Pyridine N-oxide reacts with silver(I) perchlorate monohydrate in three different stoichiometric ratios to produce three very different 1-D coordination polymers.

