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catena-Poly[silver(I)- μ -pyrazolato- κ^2 N:N']

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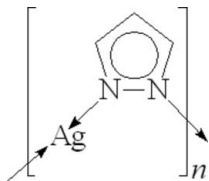
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å;
R factor = 0.041; wR factor = 0.066; data-to-parameter ratio = 17.3.

The title compound, $[\text{Ag}(\text{C}_3\text{H}_3\text{N}_2)]_n$, has an infinite helical chain structure in which each pyrazolate group bridges two Ag^{I} atoms related by a 2_1 axis with an intrachain $\text{Ag} \cdots \text{Ag}$ separation of 3.3718 (7) Å. Each Ag^{I} center is linearly coordinated by two N atoms [$\text{N}-\text{Ag}-\text{N}$ angle = 169.98 (14)°]. The chains are held together by interchain $\text{Ag} \cdots \text{Ag}$ interactions [3.2547 (6) Å], forming a two-dimensional layer. The X-ray single-crystal diffraction result is consonant with that of the *ab initio* X-ray powder diffraction reported by Masciocchi, Moret, Cairati, Sironi, Ardizzoia & La Monica [*J. Am. Chem. Soc.* (1994), **116**, 7668–7676], with only minor deviations of structural parameters.

Related literature

For related literature, see: Masciocchi *et al.* (1994).

Experimental

Crystal data

$[\text{Ag}(\text{C}_3\text{H}_3\text{N}_2)]$	$V = 830.8$ (3) Å ³
$M_r = 174.94$	$Z = 8$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
$a = 6.4084$ (13) Å	$\mu = 4.66$ mm ⁻¹
$b = 6.4989$ (13) Å	$T = 293$ (2) K
$c = 19.948$ (4) Å	$0.40 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	7019 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 1998)	951 independent reflections
$T_{\text{min}} = 0.340$, $T_{\text{max}} = 0.400$	778 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.044$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$	55 parameters
$wR(F^2) = 0.066$	H-atom parameters constrained
$S = 1.26$	$\Delta\rho_{\text{max}} = 0.38$ e Å ⁻³
951 reflections	$\Delta\rho_{\text{min}} = -0.55$ e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

$\text{Ag1}-\text{N1}$	2.070 (3)	$\text{Ag1} \cdots \text{Ag1}^{\text{ii}}$	3.2547 (6)
$\text{Ag1}-\text{N2}^{\text{i}}$	2.063 (4)	$\text{Ag1} \cdots \text{Ag1}^{\text{i}}$	3.3718 (7)
$\text{N2}^{\text{i}}-\text{Ag1}-\text{N1}$	169.98 (14)		

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *S SAINT* (Bruker, 1998); data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; 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: BQ2042).

References

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supplementary materials

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catena-Poly[silver(I)- μ -pyrazolato- κ^2 N:N']

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Comment

The synthesis and structure of silver(I)-pyrazolate, i.e. the title compound, $[\text{Ag}(\text{C}_3\text{H}_3\text{N}_2)]_n$ (I) has been reported by Masciocchi et al. (1994). In this work, the crystal structure was determined by the Ab-initio X-ray powder diffraction method and refined with the Rietveld technique in the space group of Pbc_a with $a = 6.5295$ (4), $b = 20.059$ (2) and $c = 6.4675$ (4) %Å. The result is almost consistent with the structural determination by the single-crystal diffraction reported herein, with only minor structure parameter deviations.

Compound (I) has an infinite helical chain structure, in which each pyrazolate group bridges two Ag(I) atoms related by a 2_1 axis and each Ag(I) is linearly coordinated by two N atoms from distinct pyrazolate moieties with the N-Ag-N angle of 169.98 (14) °, being larger than reported 165.5 (1) °, (Figure 1). The bond distances and angles are listed in Table 1. The torsion angle of Ag(1)-N(1)-N(2)-Ag(1B) is 22.6 (4) ° and the dihedral angle between two pyrazoly rings around one Ag(I) center is 60.3 (2) °. Furthermore, such chains are linked by interchain Ag—Ag interactions to form a 2D layer (Figure 2). The intrachain and interaction Ag—Ag separations are 3.3718 (7) [Ag(1)-Ag(1A)] and 3.2547 (6) Å [Ag(1)-Ag(1B)], respectively, which are comparable well with those reported [3.40 (1) and 3.273 (1) Å, respectively].

Experimental

AgNO₃ (85 mg, 0.5 mmol) and pyrazole (34 mg, 0.5 mmol) were dissolved in ammonium hydroxide (20%, 10 ml). The solution was filtered and filtrate was allowed to stand for 15 days. Colorless crystals of (I) were collected, in about 50% yield.

Refinement

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

Figures

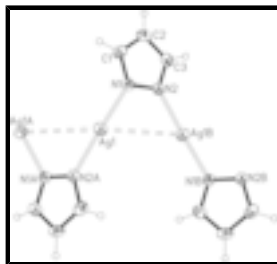


Fig. 1. Displacement ellipsoid plot (30% probability) of (I). [symmetry codes: (A) $x - 1/2, 1/2 - y, 2 - z$; (B) $x + 1/2, 1/2 - y, 2 - z$]

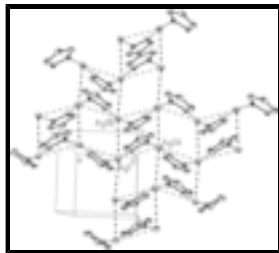


Fig. 2. Two-dimensional network in (I) linked by Ag—Ag interactions. [symmetry codes: (A) $x + 1/2, 1/2 - y, 2 - z$; (B) $1/2 - x, y - 1/2, z$]

catena-Poly[silver(I)- μ -pyrazolato- κ^2 N:N']

Crystal data

[Ag(C₃H₃N₂)]

$M_r = 174.94$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 6.4084$ (13) Å

$b = 6.4989$ (13) Å

$c = 19.948$ (4) Å

$V = 830.8$ (3) Å³

$Z = 8$

$F_{000} = 656$

$D_x = 2.797$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 6390 reflections

$\theta = 3.1$ – 27.7°

$\mu = 4.66$ mm⁻¹

$T = 293$ (2) K

Block, colorless

$0.40 \times 0.20 \times 0.20$ mm

Data collection

Bruker Smart CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

φ and ω scan

Absorption correction: multi-scan (SADABS; Bruker, 1998)

$T_{\min} = 0.340$, $T_{\max} = 0.400$

7019 measured reflections

951 independent reflections

778 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.044$

$\theta_{\max} = 27.5^\circ$

$\theta_{\min} = 3.8^\circ$

$h = -8 \rightarrow 7$

$k = -8 \rightarrow 8$

$l = -25 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.066$

$S = 1.26$

951 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0145P)^2 + 0.695P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 0.38$ e Å⁻³

55 parameters

$$\Delta\rho_{\min} = -0.55 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

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	0.23552 (5)	0.32951 (6)	1.004623 (15)	0.04206 (16)
N1	0.3965 (5)	0.3598 (6)	0.91516 (17)	0.0345 (9)
N2	0.5800 (5)	0.2552 (6)	0.90915 (17)	0.0364 (9)
C1	0.3418 (7)	0.4213 (7)	0.8548 (2)	0.0403 (12)
H1A	0.2217	0.4958	0.8450	0.048*
C2	0.4861 (7)	0.3606 (7)	0.8087 (2)	0.0448 (12)
H2A	0.4851	0.3836	0.7627	0.054*
C3	0.6325 (7)	0.2581 (7)	0.8457 (2)	0.0425 (12)
H3A	0.7528	0.1986	0.8282	0.051*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0444 (3)	0.0411 (3)	0.0407 (2)	-0.00048 (16)	0.00707 (18)	-0.00123 (16)
N1	0.038 (2)	0.032 (2)	0.033 (2)	0.0036 (18)	0.0039 (16)	-0.0005 (17)
N2	0.037 (2)	0.037 (2)	0.036 (2)	0.0002 (17)	0.0017 (17)	-0.0002 (18)
C1	0.048 (3)	0.026 (3)	0.047 (3)	0.006 (2)	-0.009 (2)	0.002 (2)
C2	0.062 (3)	0.040 (3)	0.033 (3)	-0.010 (3)	0.002 (2)	0.003 (2)
C3	0.044 (3)	0.036 (3)	0.048 (3)	-0.001 (2)	0.011 (2)	-0.006 (2)

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

Ag1—N1	2.070 (3)	N2—C3	1.310 (5)
Ag1—N2 ⁱ	2.063 (4)	C1—C2	1.362 (6)
Ag1—Ag1 ⁱⁱ	3.2547 (6)	C1—H1A	0.9300
Ag1—Ag1 ⁱ	3.3718 (7)	C2—C3	1.367 (6)
N1—C1	1.316 (5)	C2—H2A	0.9300
N1—N2	1.364 (5)	C3—H3A	0.9300
N2 ⁱ —Ag1—N1	169.98 (14)	C1—N1—N2	107.5 (3)

supplementary materials

N2 ⁱ —Ag1—Ag1 ⁱⁱ	76.18 (10)	C1—N1—Ag1	133.2 (3)
N1—Ag1—Ag1 ⁱⁱ	93.82 (10)	N2—N1—Ag1	117.3 (3)
N2 ⁱ —Ag1—Ag1 ⁱⁱⁱ	107.09 (10)	C3—N2—N1	107.4 (3)
N1—Ag1—Ag1 ⁱⁱⁱ	82.91 (10)	C3—N2—Ag1 ^{iv}	133.3 (3)
Ag1 ⁱⁱ —Ag1—Ag1 ⁱⁱⁱ	173.46 (2)	N1—N2—Ag1 ^{iv}	118.5 (2)
N2 ⁱ —Ag1—Ag1 ⁱ	60.31 (10)	N1—C1—C2	110.4 (4)
N1—Ag1—Ag1 ⁱ	117.09 (10)	N1—C1—H1A	124.8
Ag1 ⁱⁱ —Ag1—Ag1 ⁱ	75.415 (18)	C2—C1—H1A	124.8
Ag1 ⁱⁱⁱ —Ag1—Ag1 ⁱ	111.111 (17)	C1—C2—C3	104.1 (4)
N2 ⁱ —Ag1—Ag1 ^{iv}	115.02 (10)	C1—C2—H2A	128.0
N1—Ag1—Ag1 ^{iv}	60.57 (10)	C3—C2—H2A	128.0
Ag1 ⁱⁱ —Ag1—Ag1 ^{iv}	68.889 (17)	N2—C3—C2	110.6 (4)
Ag1 ⁱⁱⁱ —Ag1—Ag1 ^{iv}	104.585 (18)	N2—C3—H3A	124.7
Ag1 ⁱ —Ag1—Ag1 ^{iv}	143.72 (3)	C2—C3—H3A	124.7
N2 ⁱ —Ag1—N1—C1	108.9 (8)	C1—N1—N2—C3	0.7 (5)
Ag1 ⁱⁱ —Ag1—N1—C1	112.0 (4)	Ag1—N1—N2—C3	166.6 (3)
Ag1 ⁱⁱⁱ —Ag1—N1—C1	-73.6 (4)	C1—N1—N2—Ag1 ^{iv}	171.5 (3)
Ag1 ⁱ —Ag1—N1—C1	36.5 (5)	Ag1—N1—N2—Ag1 ^{iv}	-22.6 (4)
Ag1 ^{iv} —Ag1—N1—C1	175.2 (5)	N2—N1—C1—C2	-0.3 (5)
N2 ⁱ —Ag1—N1—N2	-52.6 (9)	Ag1—N1—C1—C2	-163.1 (3)
Ag1 ⁱⁱ —Ag1—N1—N2	-49.4 (3)	N1—C1—C2—C3	-0.1 (6)
Ag1 ⁱⁱⁱ —Ag1—N1—N2	124.9 (3)	N1—N2—C3—C2	-0.8 (5)
Ag1 ⁱ —Ag1—N1—N2	-124.9 (3)	Ag1 ^{iv} —N2—C3—C2	-169.6 (3)
Ag1 ^{iv} —Ag1—N1—N2	13.7 (2)	C1—C2—C3—N2	0.6 (6)

Symmetry codes: (i) $x-1/2, -y+1/2, -z+2$; (ii) $-x+1/2, y-1/2, z$; (iii) $-x+1/2, y+1/2, z$; (iv) $x+1/2, -y+1/2, -z+2$.

Fig. 1

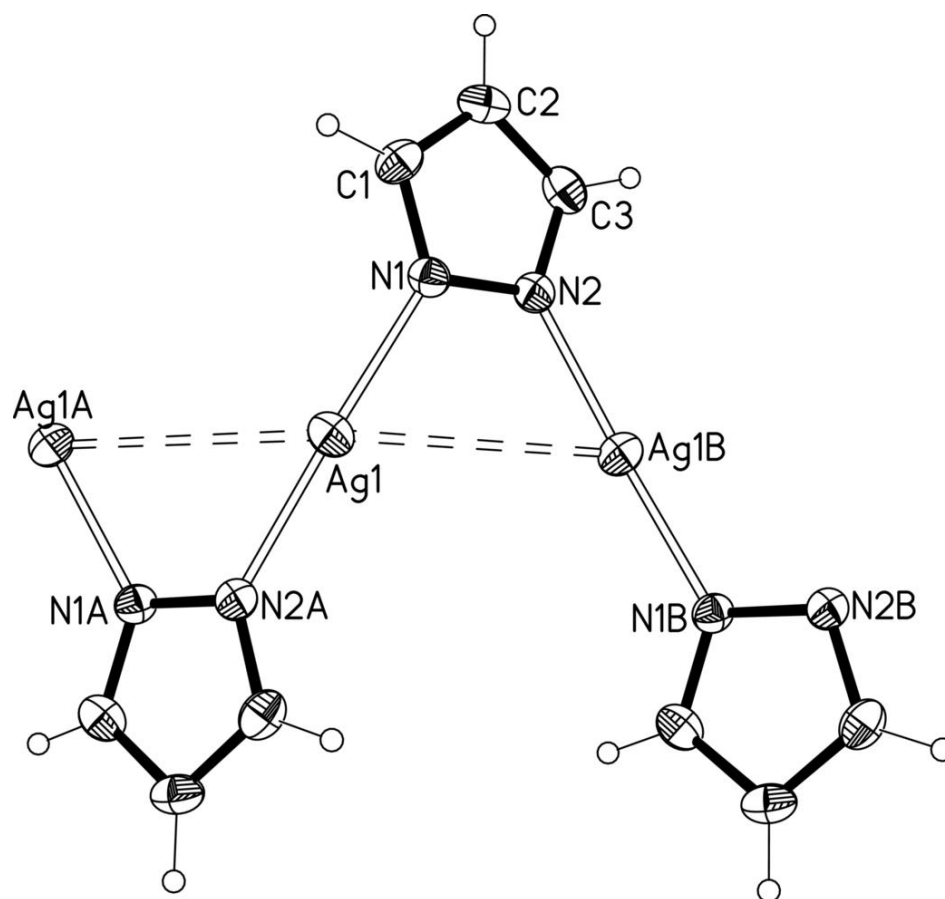


Fig. 2

