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catena-Poly[[(acetonitrile-κN)silver(I)]-di-μ-trifluoromethanesulfonato-$\kappa^4 O:O'$-[(acetonitrile-κN)silver(I)]-μ-tetraphenyldiphosphine-$\kappa^2 P:P'$]

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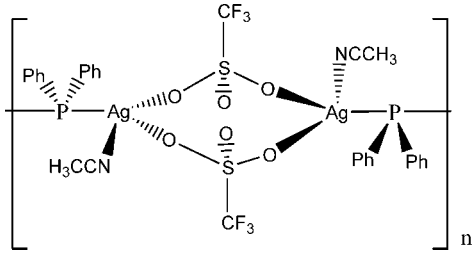
Key indicators: single-crystal X-ray study; T = 105 K; mean σ(C–C) = 0.006 Å; R factor = 0.040; wR factor = 0.112; data-to-parameter ratio = 19.8.

The silver(I)-based title polymer, $[Ag_2(CF_3SO_3)_2(C_{28}H_{26}N_2P_2)]_n$, has been synthesized and structurally characterized. The compound shows the formation of a centrosymmetric eight-membered Ag/O/S/O/Ag/O/S/O ring situated around an inversion center, with the Ag^I ions bridged by two trifluoromethanesulfonate ions in a staggered conformation. The rings are then linked by tetraphenyldiphosphine ligands into a one-dimensional polymer. The rings have an Ag···Ag separation of 5.419 (5) Å and are linked by a 2.2206 (15) Å P—P bond. All Ag^I ions have pseudo-tetrahedral geometry, with a coordination environment consisting of two O atoms from two symmetry-related trifluoromethanesulfonate ions, a P atom from a phosphine ligand and an N atom from an acetonitrile ligand.

Related literature

For the preparation of the diphosphine ligand, see: Klausmeyer *et al.* (2004).



Experimental

Crystal data

$[Ag_2(CF_3SO_3)_2(C_{28}H_{26}N_2P_2)]$
M_r = 483.16
Monoclinic, $P2_1/n$
a = 10.1128 (2) Å
b = 17.4753 (3) Å
c = 10.5053 (2) Å
β = 102.599 (1)°
V = 1811.84 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.36 mm^{-1}
T = 105 (2) K
0.21 × 0.17 × 0.10 mm

Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.760, T_{max} = 0.873
28681 measured reflections
4502 independent reflections
3588 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.112
S = 1.04
4502 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *APEX2* (Bruker, 2003); cell refinement: *APEX2* (Bruker, 2003); data reduction: *SAINT-Plus* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2003); software used to prepare material for publication: *SHELXTL*.

The Bruker X8 APEX diffractometer was purchased with funds received from the National Science Foundation Major Research Instrumentation Program, grant No. CHE-0321214. K K thanks the Robert A. Welch Foundation for support (grant No. AA-1508).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2125).

References

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Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2007). E**63**, m2395 [**doi:10.1107/S1600536807040597**]

catena-Poly[[(acetonitrile-κN)silver(I)]-di-μ-trifluoromethanesulfonato-$\kappa^4 O:O'$-[(acetonitrile-κN)silver(I)]-μ-tetraphenyldiphosphine-$\kappa^2 P:P'$]

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Comment

The title polymer contains Ag^I ions in pseudo tetrahedral geometry bridged by a rigid bidentate diphosphine (DPP) ligand (Klausmeyer *et al.*, 2004). The distorted tetrahedral angles range from 140.60 (10) to 86.61 (13)°. The Ag^I ion is stabilized by the soft donor phosphorus, from DPP, and 2 hard oxo ligands, from trifluoromethanesulfonate. The structure contains an 8-membered ring containing the silvers and the anions (Fig. 1). This ring is situated on an inversion center. The rings are then linked by the diphosphine ligand through the silvers into a one-dimensional polymer which boasts a 'Ring—P—P—Ring' design resembling a staircase (Fig. 2). The extended packing structure indicates that F atoms on the trifluoromethanesulfonate ion form a 3.186 (4) Å interaction with a C—H group on a neighboring 'staircase' polymer.

Experimental

The DPP ligand is an impurity formed in the synthesis of PCP-31 (Klausmeyer *et al.*, 2004). Then combined with an equivalent of Ag(I)OTF in 10 ml CH_3CN, a clear colorless solution results. Colorless blocks of the title compound were obtained by the slow diffusion of ether into a CH_3CN solution containing the complex at 278 K.

Refinement

H atoms were included in calculated positions and refined as riding on their parent atoms, with C—H bond lengths fixed to 0.95 (aromatic CH), or 0.98 Å (methyl CH_3). Isotropic displacement parameters were fixed for H atoms: U_{iso}(H) = 1.5U_{eq}(C15) for the methyl group and U_{iso}(H) = 1.2U_{eq}(carrier C) for phenyl groups. In the last difference map, the highest residual peak, 1.47 e.Å^{-3}, is found at 1.03 Å from Ag1.

Figures

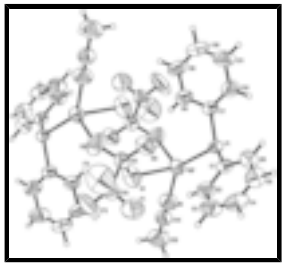

Fig. 1. Thermal ellipsoid plot of the monomer unit of the title compound. Displacement ellipsoids are shown at the 30% probability level and the labeling scheme is given for the asymmetric unit. Non-labeled atoms are generated by symmetry code $1-x, 2-y, -z$.

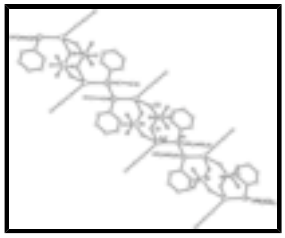

Fig. 2. Ball-and-Stick diagram displaying the 'Ring-P—P-Ring' pattern observed in the extended structure of the title polymer. H atoms have been omitted for clarity.

catena-Poly[[(acetonitrile-κ_N_)silver(I)]-di-μ- trifluoromethanesulfonato-κ^4_O_:_O_'-[(acetonitrile-κ_N_)silver(I)]-μ-tetraphenyldiphosphine-κ^2_P_:_P_']

Crystal data

$[Ag_2(CF_3SO_3)_2(C_{28}H_{26}N_2P_2)]$	F_{000} = 956
M_r = 483.16	D_x = 1.771 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 8448 reflections
a = 10.1128 (2) Å	θ = 2.3–28.0º
b = 17.4753 (3) Å	μ = 1.36 mm^{-1}
c = 10.5053 (2) Å	T = 105 (2) K
β = 102.599 (1)º	Rod, colourless
V = 1811.84 (6) Å^3	0.21 × 0.17 × 0.10 mm
Z = 4	

Data collection

Bruker X8 APEX diffractometer	4502 independent reflections
Radiation source: fine-focus sealed tube	3588 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
T = 105(2) K	θ_{max} = 28.3º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→13
T_{min} = 0.760, T_{max} = 0.873	k = −23→23
28681 measured reflections	l = −14→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H-atom parameters constrained
$wR(F^2)$ = 0.112	$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 1.0373P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$

4502 reflections	$\Delta\rho_{max}$ = 1.47 e Å^{-3}
227 parameters	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.61713 (3)	1.022971 (14)	0.25471 (3)	0.05425 (11)
P1	0.50042 (7)	0.96768 (4)	0.40910 (7)	0.03942 (17)
S3	0.57444 (9)	0.87763 (4)	0.03092 (8)	0.05077 (19)
C7	0.3200 (3)	0.95576 (16)	0.3394 (3)	0.0439 (6)
C8	0.2762 (4)	0.9755 (2)	0.2101 (4)	0.0623 (9)
H8	0.3393	0.9941	0.1624	0.075*
C1	0.5731 (3)	0.87625 (16)	0.4756 (3)	0.0442 (6)
C2	0.6818 (4)	0.8485 (2)	0.4273 (4)	0.0607 (8)
H2	0.7138	0.8765	0.3627	0.073*
C6	0.5306 (4)	0.83521 (19)	0.5713 (4)	0.0590 (8)
H6	0.4571	0.8535	0.6057	0.071*
C4	0.6990 (4)	0.7402 (2)	0.5701 (5)	0.0736 (11)
H4	0.7425	0.6938	0.6028	0.088*
C5	0.5950 (4)	0.7669 (2)	0.6178 (4)	0.0667 (10)
H5	0.5649	0.7390	0.6838	0.080*
C12	0.2260 (4)	0.9294 (3)	0.4062 (4)	0.0729 (11)
H12	0.2548	0.9145	0.4949	0.087*
C13	0.6961 (5)	0.8024 (3)	0.0321 (4)	0.0731 (11)
C9	0.1401 (5)	0.9684 (3)	0.1490 (5)	0.0819 (13)
H9	0.1112	0.9812	0.0593	0.098*
C11	0.0889 (4)	0.9245 (3)	0.3452 (5)	0.0830 (13)
H11	0.0245	0.9081	0.3932	0.100*
C3	0.7430 (4)	0.7802 (3)	0.4734 (5)	0.0757 (11)
H3	0.8154	0.7607	0.4387	0.091*
C10	0.0478 (4)	0.9433 (2)	0.2168 (5)	0.0735 (12)
H10	−0.0450	0.9388	0.1745	0.088*
F2	0.7758 (4)	0.8166 (3)	−0.0462 (4)	0.1362 (14)
F3	0.6357 (4)	0.73743 (16)	−0.0037 (4)	0.1363 (15)
F1	0.7723 (3)	0.7912 (2)	0.1497 (3)	0.1225 (12)
O2	0.4961 (3)	0.85106 (17)	0.1200 (3)	0.0738 (7)
O1	0.6587 (3)	0.94232 (16)	0.0761 (3)	0.0799 (8)
N1	0.8058 (3)	1.0888 (2)	0.2480 (4)	0.0730 (9)
C14	0.8980 (4)	1.1225 (2)	0.2413 (4)	0.0650 (9)
O3	0.5061 (4)	0.88004 (19)	−0.1030 (3)	0.0885 (10)
C15	1.0188 (5)	1.1660 (3)	0.2352 (6)	0.0971 (16)
H15A	1.0059	1.2196	0.2572	0.146*
H15B	1.0353	1.1630	0.1468	0.146*
H15C	1.0966	1.1447	0.2974	0.146*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.05592 (17)	0.05287 (16)	0.05747 (17)	−0.00550 (10)	0.02006 (12)	0.00355 (10)
P1	0.0370 (4)	0.0361 (3)	0.0438 (4)	−0.0028 (3)	0.0059 (3)	0.0003 (3)
S3	0.0570 (4)	0.0431 (4)	0.0537 (4)	0.0051 (3)	0.0155 (4)	0.0008 (3)
C7	0.0388 (14)	0.0346 (13)	0.0553 (17)	−0.0022 (11)	0.0039 (12)	−0.0029 (12)
C8	0.0480 (18)	0.073 (2)	0.062 (2)	0.0061 (16)	0.0032 (16)	0.0082 (17)
C1	0.0421 (14)	0.0360 (13)	0.0508 (16)	0.0020 (11)	0.0018 (12)	0.0001 (12)
C2	0.0521 (18)	0.063 (2)	0.066 (2)	0.0107 (16)	0.0090 (16)	0.0091 (17)
C6	0.064 (2)	0.0435 (16)	0.073 (2)	0.0015 (14)	0.0228 (18)	0.0042 (15)
C4	0.063 (2)	0.0495 (19)	0.097 (3)	0.0097 (17)	−0.007 (2)	0.016 (2)
C5	0.075 (2)	0.0462 (18)	0.077 (2)	−0.0031 (17)	0.011 (2)	0.0162 (17)
C12	0.056 (2)	0.096 (3)	0.063 (2)	−0.024 (2)	0.0046 (17)	0.011 (2)
C13	0.079 (3)	0.073 (3)	0.072 (2)	0.023 (2)	0.027 (2)	0.002 (2)
C9	0.056 (2)	0.102 (4)	0.073 (3)	0.009 (2)	−0.016 (2)	0.010 (2)
C11	0.049 (2)	0.094 (3)	0.104 (3)	−0.023 (2)	0.013 (2)	0.012 (3)
C3	0.059 (2)	0.073 (3)	0.094 (3)	0.0232 (19)	0.014 (2)	0.009 (2)
C10	0.0448 (19)	0.062 (2)	0.098 (3)	0.0001 (16)	−0.017 (2)	−0.002 (2)
F2	0.122 (3)	0.182 (4)	0.134 (3)	0.054 (2)	0.090 (2)	0.022 (3)
F3	0.177 (4)	0.0536 (17)	0.182 (4)	0.0202 (19)	0.046 (3)	−0.0193 (19)
F1	0.102 (2)	0.163 (3)	0.099 (2)	0.071 (2)	0.0145 (17)	0.024 (2)
O2	0.0638 (16)	0.0796 (18)	0.0868 (19)	−0.0078 (14)	0.0355 (15)	−0.0007 (15)
O1	0.102 (2)	0.0586 (15)	0.090 (2)	−0.0251 (15)	0.0457 (18)	−0.0175 (14)
N1	0.0616 (19)	0.071 (2)	0.089 (2)	−0.0154 (16)	0.0215 (17)	−0.0050 (18)
C14	0.059 (2)	0.068 (2)	0.069 (2)	−0.0121 (17)	0.0172 (18)	−0.0080 (18)
O3	0.111 (2)	0.081 (2)	0.0627 (17)	0.0280 (18)	−0.0042 (16)	0.0076 (14)
C15	0.068 (3)	0.105 (4)	0.123 (4)	−0.028 (3)	0.031 (3)	−0.007 (3)

Geometric parameters (Å, °)

Ag1—N1	2.242 (3)	C4—C5	1.344 (6)
Ag1—P1	2.4068 (8)	C4—C3	1.383 (6)
Ag1—O1	2.455 (3)	C4—H4	0.9500
Ag1—O3^{i}	2.470 (3)	C5—H5	0.9500
P1—C7	1.824 (3)	C12—C11	1.398 (5)
P1—C1	1.832 (3)	C12—H12	0.9500
P1—P1^{ii}	2.2206 (15)	C13—F2	1.295 (5)
S3—O3	1.427 (3)	C13—F3	1.305 (5)
S3—O2	1.429 (3)	C13—F1	1.320 (5)
S3—O1	1.433 (3)	C9—C10	1.365 (7)
S3—C13	1.799 (4)	C9—H9	0.9500
C7—C8	1.378 (5)	C11—C10	1.362 (7)
C7—C12	1.378 (5)	C11—H11	0.9500
C8—C9	1.392 (6)	C3—H3	0.9500
C8—H8	0.9500	C10—H10	0.9500
C1—C6	1.378 (5)	N1—C14	1.118 (5)

C1—C2	1.394 (5)	C14—C15	1.452 (6)
C2—C3	1.383 (5)	O3—Ag1^{i}	2.470 (3)
C2—H2	0.9500	C15—H15A	0.9800
C6—C5	1.396 (5)	C15—H15B	0.9800
C6—H6	0.9500	C15—H15C	0.9800
N1—Ag1—P1	140.60 (10)	C3—C4—H4	119.9
N1—Ag1—O1	88.97 (11)	C4—C5—C6	120.6 (4)
P1—Ag1—O1	118.38 (7)	C4—C5—H5	119.7
N1—Ag1—O3^{i}	86.61 (13)	C6—C5—H5	119.7
P1—Ag1—O3^{i}	117.23 (9)	C7—C12—C11	120.8 (4)
O1—Ag1—O3^{i}	92.72 (11)	C7—C12—H12	119.6
C7—P1—C1	109.67 (13)	C11—C12—H12	119.6
C7—P1—P1^{ii}	102.26 (11)	F2—C13—F3	107.5 (4)
C1—P1—P1^{ii}	101.08 (11)	F2—C13—F1	107.8 (4)
C7—P1—Ag1	111.71 (11)	F3—C13—F1	106.4 (4)
C1—P1—Ag1	113.08 (11)	F2—C13—S3	112.0 (3)
P1^{ii}—P1—Ag1	118.02 (5)	F3—C13—S3	110.9 (3)
O3—S3—O2	115.9 (2)	F1—C13—S3	111.9 (3)
O3—S3—O1	115.4 (2)	C10—C9—C8	120.6 (4)
O2—S3—O1	114.45 (17)	C10—C9—H9	119.7
O3—S3—C13	102.2 (2)	C8—C9—H9	119.7
O2—S3—C13	103.52 (19)	C10—C11—C12	119.8 (4)
O1—S3—C13	102.6 (2)	C10—C11—H11	120.1
C8—C7—C12	118.5 (3)	C12—C11—H11	120.1
C8—C7—P1	116.4 (3)	C4—C3—C2	120.1 (4)
C12—C7—P1	125.0 (3)	C4—C3—H3	119.9
C7—C8—C9	120.3 (4)	C2—C3—H3	119.9
C7—C8—H8	119.8	C11—C10—C9	119.9 (4)
C9—C8—H8	119.8	C11—C10—H10	120.0
C6—C1—C2	118.7 (3)	C9—C10—H10	120.0
C6—C1—P1	124.7 (2)	S3—O1—Ag1	121.18 (17)
C2—C1—P1	116.6 (3)	C14—N1—Ag1	177.9 (4)
C3—C2—C1	120.1 (4)	N1—C14—C15	178.9 (5)
C3—C2—H2	120.0	S3—O3—Ag1^{i}	136.42 (19)
C1—C2—H2	120.0	C14—C15—H15A	109.5
C1—C6—C5	120.3 (3)	C14—C15—H15B	109.5
C1—C6—H6	119.9	H15A—C15—H15B	109.5
C5—C6—H6	119.9	C14—C15—H15C	109.5
C5—C4—C3	120.1 (3)	H15A—C15—H15C	109.5
C5—C4—H4	119.9	H15B—C15—H15C	109.5

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*; (ii) −*x*+1, −*y*+2, −*z*+1.

Fig. 1

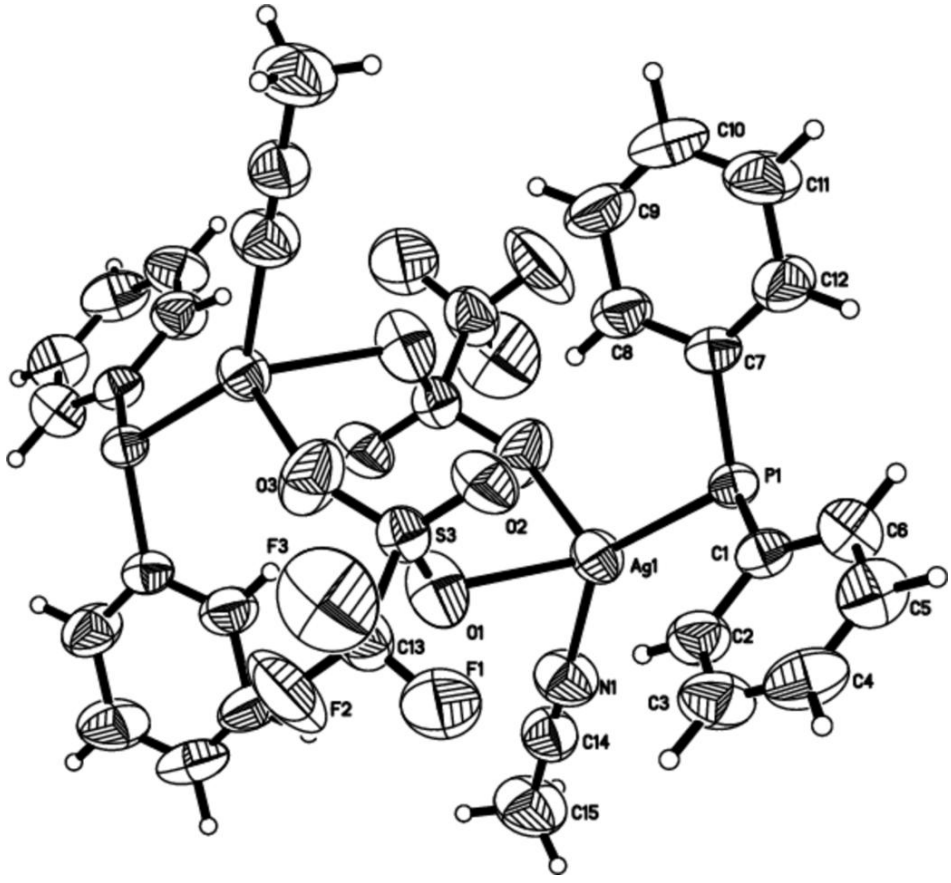
C10
C11
C9
C12
C8
C7
O3
P1
S3
O2
F3
Ag1
C1
C6
O1
C5
C13
C2
F1
N1
C4
C3
F2
C14
C15

Fig. 2