Crystal structure of tetrakis(triphenylphospine-κP)silver(I) trifluoromethanesulfonate, $[Ag(P(C_6H_5)_3)_4][SO_3CF_3]$

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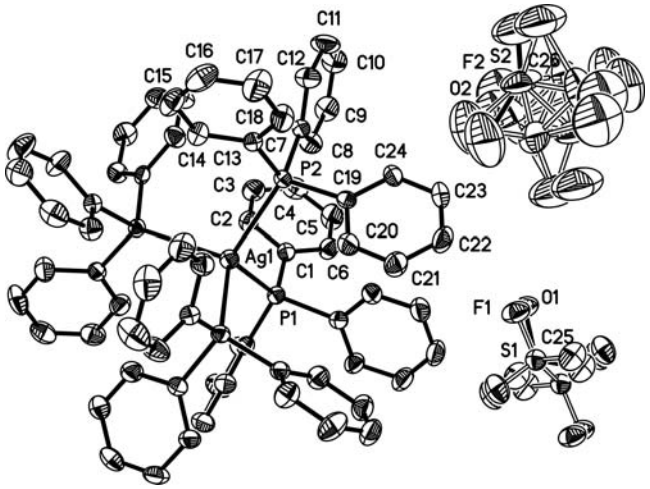

Abstract

$C_{73}H_{60}AgF_3O_3P_4S$, trigonal, $R\bar{3}$ (no. 148), a = 14.324(2) Å, c = 51.876(4) Å, V = 9217.3 Å^3, Z = 6, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.178, T = 298 K.

Source of material

The title compound was obtained by the reaction of silver(I) trifluoromethanesulfonate (AgOTf) and triphenlyphosphine (PPh_3) (molar ratio 1:1) in the presence of 1,2-di(2-pyridyl)ethylene (0.1 mmol) in the mixed solvent CH_3OH/CH_2Cl_2 (10 ml, 1:1, *v*/*v*) for 6 hours at ambient temperature. The insoluble residue was removed by filtration, and the filtrate was evaporated slowly at room temperature for about one week to yield colorless crystals. Crystals suitable for single crystal X-ray diffraction were selected directly from the sample as prepared.

Experimental details

In each independent unit, S1 and C25 occupy the same position with the total occupancy 1/3. The occupancy of S1 and C25 is determined as 1/6 since the ratio of S1/C25 must be 1:1. S2 and C26 also occupy the same position with total occupancy 1. The position of S2 and C26 generate 6 positions through symmetry operation, so S2 and C26 are disordered and the occupancy of each atom is determined as 1/6.

Discussion

Great interest has been focused on the silver(I) complexes containing triphenlyphosphine phosphorous and heterocyclic nitrogen ligands because of their various structures and their potential applications as catalysts and lunminescence materials [1-3]. In the course of our work on the syntheses of Ag(I) compounds, we find that some heterocylic N ligands play an important role in the formation of products of mixed P and N-ligands with special structure. For example, $[Ag_4SCN_4dppm_2]$ [4] and $[AgSCNdppm]_2$ [5] were obtained in presence of quinoline and phenathroline, respectively; $[AgClO_4(PPh_3)_3]$ [6], $[AgClO_4(PPh_3)_3(MeOH)]$ [7] and $[Ag(PPh_3)(CH_3COO)]_2 \cdot H_2O \cdot CH_3OH$ [8] were prepared in presence of 2-aminopyrimidine. Attempting to prepare Ag(I) complex of 1,2-di(2-pyridyl)ethylene and PPh_3, we unexpectedly obtained the title compound.

The title crystal structure consists of one $[Ag(PPh_3)_4]$ cation and one disordered trifuoromethanesulfonate anion in the asymmetric unit. The Ag is coordinated by four P atoms from four PPh_3 ligands in a slightly distorted tetrahedral environment. The d(Ag—P) are in the range of 2.624(2) - 2.654(1) Å, and are longer than those in $[AgClO_4(PPh_3)_3]$ (2.5047 - 2.564(1) Å), $[AgClO_4(PPh_3)_2(MeOH)]$ (2.430(8) - 2.428(1) Å) and $[Ag(C_{12}H_8N_2)(C_{18}H_{15}P)][CF_3SO_3]$ (2.29(2) Å) [9]. The P–Ag–P angles are in the range of 109.45(3)° - 109.50(3)°, and are smaller than those in $[AgClO_4(PPh_3)_3]$ (114.70° - 119.17°), $[Ag(PPh_3)(CH_3COO)]_2 \cdot H_2O \cdot CH_3OH$ (128.13° - 128.27°) and $[AgClO_4(PPh_3)_2(MeOH)]$ (133.5°).

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.41 × 0.44 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.25 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15574, 3629
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2393
$N(param)_{refined}$:	288
Programs:	SHELXS-97, SHELXL-97 [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	18*f*	0.8835	0.3955	0.6238	0.06
H(3)	18*f*	1.0464	0.5079	0.6421	0.07
H(4)	18*f*	1.0587	0.6123	0.6768	0.08
H(5)	18*f*	0.9049	0.6007	0.6937	0.09
H(6)	18*f*	0.7397	0.4886	0.6749	0.06
H(8)	18*f*	0.8382	0.5797	0.6115	0.07
H(9)	18*f*	0.9848	0.7255	0.6294	0.11
H(10)	18*f*	1.0832	0.8790	0.6061	0.06
H(11)	18*f*	1.0342	0.8874	0.5640	0.07
H(12)	18*f*	0.8884	0.7404	0.5456	0.06

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(14)	18*f*	0.7998	0.4512	0.5246	0.08
H(15)	18*f*	0.8317	0.4807	0.4813	0.12
H(16)	18*f*	0.7849	0.5913	0.4602	0.09
H(17)	18*f*	0.7092	0.6750	0.4819	0.11
H(18)	18*f*	0.6771	0.6465	0.5255	0.05

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(20)	18*f*	0.4980	0.4234	0.5618	0.09
H(21)	18*f*	0.3628	0.4568	0.5723	0.08
H(22)	18*f*	0.4027	0.6182	0.5913	0.07
H(23)	18*f*	0.5790	0.7488	0.5971	0.07
H(24)	18*f*	0.7161	0.7159	0.5865	0.06

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	6*c*		⅔	⅓	0.58185(1)	0.0426(3)	U_{11}	0.0430(4)	½U_{11}	0	0
P(1)	6*c*		⅔	⅓	0.63244(4)	0.0402(7)	U_{11}	0.032(1)	½U_{11}	0	0
P(2)	18*f*		0.7232(1)	0.52931(9)	0.56482(2)	0.0386(7)	0.0326(7)	0.0404(7)	0.0186(6)	0.0022(5)	0.0024(5)
F(1)	18*f*	0.50	0.432(4)	0.725(4)	0.6390(8)	0.07(1)	0.08(2)	0.05(2)	0.02(1)	0.02(1)	0.001(9)
F(2)	18*f*	0.50	0.111(8)	0.997(9)	0.517(3)	0.16(5)	0.23(7)	0.17(1)	0.12(4)	–0.05(4)	0.03(5)
O(1)	18*f*	0.50	0.445(6)	0.746(5)	0.645(1)	0.07(1)	0.08(2)	0.05(2)	0.02(1)	0.02(1)	0.001(9)
O(2)	18*f*	0.50	0.08(1)	0.96(1)	0.519(4)	0.16(5)	0.23(7)	0.17(1)	0.12(4)	–0.05(4)	0.03(5)
S(1)	6*c*	0.50	⅓	⅔	0.64934(7)	0.049(1)	U_{11}	0.050(2)	½U_{11}	0	0
S(2)	18*f*	0.17	0.062(1)	1.014(5)	0.4970(8)	0.069(6)	0.13(2)	0.12(2)	0.05(1)	0.043(9)	0.09(2)
C(1)	18*f*		0.7936(4)	0.4277(4)	0.64779(9)	0.047(3)	0.041(3)	0.035(2)	0.024(2)	0.000(2)	0.002(2)
C(2)	18*f*		0.8870(4)	0.4366(4)	0.6381(1)	0.050(3)	0.056(3)	0.053(3)	0.028(3)	–0.001(3)	–0.002(3)
C(3)	18*f*		0.9844(5)	0.5040(5)	0.6488(1)	0.045(3)	0.067(4)	0.080(4)	0.027(3)	–0.004(3)	0.000(3)
C(4)	18*f*		0.9918(5)	0.5658(5)	0.6695(1)	0.048(4)	0.062(4)	0.086(5)	0.015(3)	–0.021(3)	–0.007(4)
C(5)	18*f*		0.9005(5)	0.5593(5)	0.6795(1)	0.065(4)	0.079(5)	0.073(4)	0.029(4)	–0.019(3)	–0.036(4)
C(6)	18*f*		0.8015(5)	0.4913(4)	0.6684(1)	0.048(3)	0.062(4)	0.052(3)	0.024(3)	–0.003(3)	–0.018(3)
C(7)	18*f*		0.8463(4)	0.6448(4)	0.57689(9)	0.038(3)	0.036(3)	0.046(3)	0.019(2)	0.002(2)	–0.001(2)
C(8)	18*f*		0.8774(5)	0.6419(5)	0.6017(1)	0.064(4)	0.055(4)	0.054(3)	0.029(3)	–0.007(3)	0.003(3)
C(9)	18*f*		0.9654(5)	0.7292(5)	0.6125(1)	0.062(4)	0.078(5)	0.058(4)	0.032(4)	–0.012(3)	–0.012(3)
C(10)	18*f*		1.0238(5)	0.8200(5)	0.5988(1)	0.050(4)	0.066(4)	0.084(5)	0.016(3)	–0.015(3)	–0.021(4)
C(11)	18*f*		0.9945(5)	0.8248(5)	0.5736(2)	0.064(4)	0.048(4)	0.090(5)	–0.005(3)	0.010(4)	0.000(3)
C(12)	18*f*		0.9068(5)	0.7372(5)	0.5626(1)	0.064(4)	0.054(4)	0.057(4)	0.017(3)	0.001(3)	0.005(3)
C(13)	18*f*		0.7371(4)	0.5478(4)	0.53004(9)	0.040(3)	0.038(3)	0.039(3)	0.019(2)	0.002(2)	0.000(2)
C(14)	18*f*		0.7819(4)	0.4974(5)	0.5162(1)	0.055(3)	0.061(4)	0.053(3)	0.033(3)	0.004(3)	–0.002(3)
C(15)	18*f*		0.8004(5)	0.5142(6)	0.4903(1)	0.063(4)	0.098(5)	0.053(4)	0.036(4)	0.007(3)	–0.018(4)
C(16)	18*f*		0.7726(6)	0.5801(7)	0.4779(1)	0.078(5)	0.121(6)	0.041(3)	0.043(5)	0.009(3)	0.012(4)
C(17)	18*f*		0.7276(7)	0.6298(7)	0.4906(1)	0.126(7)	0.132(7)	0.049(4)	0.092(6)	0.017(4)	0.032(4)
C(18)	18*f*		0.7091(6)	0.6131(5)	0.5167(1)	0.098(5)	0.084(5)	0.055(4)	0.066(4)	0.010(3)	0.017(3)
C(19)	18*f*		0.6215(4)	0.5658(4)	0.57298(9)	0.044(3)	0.035(3)	0.038(3)	0.020(2)	0.004(2)	0.009(2)
C(20)	18*f*		0.5150(4)	0.4896(5)	0.5690(1)	0.049(3)	0.047(3)	0.073(4)	0.023(3)	0.002(3)	–0.003(3)
C(21)	18*f*		0.4342(5)	0.5090(5)	0.5754(1)	0.045(4)	0.070(4)	0.085(4)	0.031(3)	–0.003(3)	–0.008(3)
C(22)	18*f*		0.4579(5)	0.6055(6)	0.5864(1)	0.059(4)	0.083(5)	0.077(4)	0.052(4)	0.013(3)	0.008(4)
C(23)	18*f*		0.5626(5)	0.6825(5)	0.5901(1)	0.075(5)	0.055(4)	0.077(4)	0.044(4)	0.005(3)	–0.009(3)
C(24)	18*f*		0.6447(5)	0.6631(4)	0.5837(1)	0.050(3)	0.039(3)	0.064(3)	0.023(3)	0.000(3)	–0.005(3)
C(25)	6*c*	0.50	⅓	⅔	0.64934	0.049	U_{11}	0.050	½U_{11}	0	0
C(26)	18*f*	0.17	0.062(1)	1.014(5)	0.4970(8)	0.069(6)	0.13(2)	0.12(2)	0.05(1)	0.043(9)	0.09(2)

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