# Crystal structure of bis(triphenylphospine-κ*P*)(methanol-κ*O*)(tetrafluoroborate-κ*F*)silver(I), $[Ag(PPh_3)_2(CH_3OH)BF_4]$, $C_{37}H_{34}AgBF_4OP_2$

Sen Gao[I], Qi-Ming Qiu[I], Min Liu[II], Yu-Han Jiang[I] and Qiong-Hua Jin[*, I]

[I] Department of Chemistry, Capital Normal University, Beijing 100048, P. R. China
[II] The College of Materials Science and Engineering, Beijing University of Technology, Beijing 100022, P. R. China

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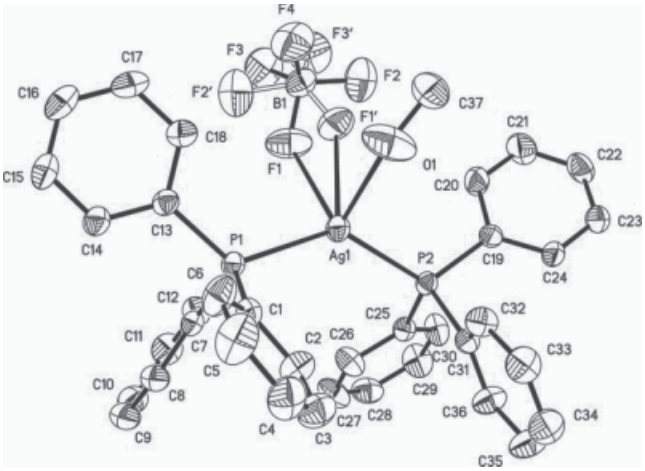


**Abstract**

$C_{37}H_{34}AgBF_4OP_2$, monoclinic, $P2_1/n$ (no. 14), $a$ = 13.748(1) Å, $b$ = 12.757(1) Å, $c$ = 19.716(2) Å, $\beta$ = 92.657(1)°, $V$ = 3454.1 Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0361, $wR_{ref}(F^2)$ = 0.1018, $T$ = 298 K.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colourless blocks, size 0.40×0.45×0.48 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 7.26 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART 1000 CCD, $\varphi$ and $\omega$ |
| $2\theta_{max}$: | 50.04° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 16819, 6077 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 4316 |
| $N(param)_{refined}$: | 444 |
| Programs: | SHELX [11], SADABS [12] |

**Source of material**

2,5-dimercapto-1,3,4-thiadiazole (0.1 mmol, 0.015 g) and triphenylphosphine ($PPh_3$, 0.4 mmol, 0.105 g) were added into the stirring solution of $AgBF_4$ (0.2 mmol, 0.039 g) in a mixture of $CH_2Cl_2$ (5ml) and $CH_3OH$ (5ml). After being stirred for 2h at room temperature, the white precipitate was filtered off. Subsequent slow evaporation of the colourless filtrate resulted in the formation of colourless crystals of the title complex $[Ag(PPh_3)_2(CH_3OH)BF_4]$. Crystals suitable for single crystal X-ray diffraction were selected directly from the sample as prepared.

**Discussion**

In recent years, reports concerned with silver(I) complexes are growing because of their interesting structures, special spectroscopic properties and potential applications in materials science [1-4]. Especially, some compounds containing silver and triphenylphosphine have been synthesized [5-7]. Herein, we report the title complex$[Ag(PPh_3)_2(CH_3OH)BF_4]$, which was unexpectedly obtained in an attempt to prepare a silver(I) complex of 2,5-dimercapto-1,3,4-thiadiazole and $PPh_3$. In the neutral complex, $[Ag(PPh_3)_2(CH_3OH)BF_4]$, the Ag(I) center is coordinated by two P atoms from two $PPh_3$ ligands, one F atom from the tetrafluoroborate anion and one O atom from the methanol ligand. The coordination geometry around the Ag(I) atom is like a distorted tetrahedron, which is confirmed by the angles P–Ag–P of 134.78(3)°, O–Ag–F of 91.48(16)°, O–Ag–P of 107.56(11)° and 105.23(12)°, P–Ag–F of 96.31(16)° and 113.14(13)°. In the title structure, important bond distances are: Ag–P (2.4268(9) Å and 2.4273(9) Å), Ag–O (2.421(3) Å) and Ag–F (2.592(5) Å and 2.70(3) Å). The Ag–P distances are slightly shorter than that in complexes $[Ag(PPh_3)_2](CF_3SO_3)$ (Ag–P = 2.4330(12) Å and 2.4321(12) Å), and $[Ag(PPh_3)_2(H_2PO_4)]\cdot 2EtOH$ (Ag–P = 2.4309(4) Å and 2.4595(4) Å) [8, 9], and is much shorter than that in complexes $[AgBr(PPh_3)_2]$ (Ag–P = 2.568(1) Å and 2.458(2) Å), $[Ag(C_{18}H_{15}P)_4]CF_3O_3S\cdot CH_2Cl_2$ (Ag–P in the range of 2.6202(17)-2.6682(17) Å), and $[Ag(P(C_6H_5)_3)_4][SO_3CF_3]$ (Ag–P in the range of 2.624(2)-2.654(1) Å) [10, 6, 7]. The angle P–Ag–P is larger than that in complexes $[Ag(PPh_3)_2](CF_3SO_3)$ (131.20(4)°), $[Ag(PPh_3)_2(H_2PO_4)]\cdot 2EtOH$ (127.671(14)°) [8, 9].

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 4*e* | 0.2421 | 0.7797 | 0.2416 | 0.180 |
| H(2) | 4*e* | 0.5174 | 0.8860 | 0.1346 | 0.081 |
| H(3) | 4*e* | 0.5509 | 1.0538 | 0.1711 | 0.096 |
| H(4) | 4*e* | 0.5953 | 1.0837 | 0.2820 | 0.103 |
| H(5) | 4*e* | 0.6020 | 0.9487 | 0.3581 | 0.117 |
| H(6) | 4*e* | 0.5675 | 0.7792 | 0.3232 | 0.089 |
| H(8) | 4*e* | 0.7142 | 0.7483 | 0.1718 | 0.064 |
| H(9) | 4*e* | 0.8311 | 0.6981 | 0.0976 | 0.080 |
| H(10) | 4*e* | 0.7952 | 0.5766 | 0.0143 | 0.082 |
| H(11) | 4*e* | 0.6433 | 0.5022 | 0.0046 | 0.086 |
| H(12) | 4*e* | 0.5257 | 0.5509 | 0.0781 | 0.072 |
| H(14) | 4*e* | 0.6821 | 0.5914 | 0.2604 | 0.069 |
| H(15) | 4*e* | 0.7074 | 0.4657 | 0.3431 | 0.081 |
| H(16) | 4*e* | 0.5777 | 0.3854 | 0.3907 | 0.086 |
| H(17) | 4*e* | 0.4229 | 0.4335 | 0.3576 | 0.087 |
| H(18) | 4*e* | 0.3941 | 0.5595 | 0.2752 | 0.069 |
| H(20) | 4*e* | 0.1463 | 0.6158 | 0.0399 | 0.085 |
| H(21) | 4*e* | –0.0152 | 0.5917 | 0.0055 | 0.109 |
| H(22) | 4*e* | –0.1041 | 0.7267 | –0.0417 | 0.085 |
| H(23) | 4*e* | –0.0356 | 0.8881 | –0.0507 | 0.074 |

* Correspondence author (e-mail: jinqh204@163.com)

**Table 2.** continued.

| Atom | Site | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|
| H(24) | 4*e* | 0.1258 | 0.9132 | –0.0191 | 0.064 |
| H(26) | 4*e* | 0.4774 | 0.7317 | –0.0053 | 0.077 |
| H(27) | 4*e* | 0.5524 | 0.6848 | –0.1029 | 0.095 |
| H(28) | 4*e* | 0.4630 | 0.6569 | –0.2011 | 0.085 |
| H(29) | 4*e* | 0.2988 | 0.6869 | –0.2058 | 0.098 |
| H(30) | 4*e* | 0.2220 | 0.7370 | –0.1092 | 0.079 |
| H(32) | 4*e* | 0.2717 | 0.9250 | 0.1374 | 0.073 |

**Table 2.** continued.

| Atom | Site | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|
| H(33) | 4*e* | 0.2731 | 1.1060 | 0.1467 | 0.088 |
| H(34) | 4*e* | 0.3106 | 1.2072 | 0.0561 | 0.099 |
| H(35) | 4*e* | 0.3453 | 1.1308 | –0.0451 | 0.104 |
| H(36) | 4*e* | 0.3419 | 0.9504 | –0.0558 | 0.080 |
| H(37A) | 4*e* | 0.1169 | 0.6907 | 0.2644 | 0.152 |
| H(37B) | 4*e* | 0.1284 | 0.6313 | 0.1956 | 0.152 |
| H(37C) | 4*e* | 0.0982 | 0.7498 | 0.1953 | 0.152 |

**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) | 4*e* | | 0.35130(2) | 0.69302(2) | 0.13376(1) | 0.0449(2) | 0.0537(2) | 0.0455(2) | –0.0001(1) | –0.0070(1) | 0.0058(2) |
| F(1) | 4*e* | 0.866 | 0.3162(5) | 0.4935(4) | 0.1364(2) | 0.137(5) | 0.085(4) | 0.121(3) | –0.049(4) | 0.028(3) | –0.007(3) |
| F(2) | 4*e* | 0.866 | 0.1584(4) | 0.4604(5) | 0.1183(2) | 0.117(4) | 0.135(6) | 0.119(3) | 0.023(4) | –0.031(3) | 0.007(3) |
| F(3) | 4*e* | 0.866 | 0.2617(5) | 0.3318(3) | 0.1417(3) | 0.133(5) | 0.047(2) | 0.166(5) | 0.007(3) | 0.018(4) | –0.021(3) |
| F(1') | 4*e* | 0.134 | 0.241(3) | 0.518(2) | 0.131(2) | 0.12(3) | 0.06(2) | 0.13(2) | –0.01(2) | 0.01(2) | 0.02(2) |
| F(2') | 4*e* | 0.134 | 0.331(3) | 0.383(3) | 0.174(2) | 0.13(3) | 0.13(3) | 0.14(3) | 0.03(2) | –0.02(2) | –0.01(2) |
| F(3') | 4*e* | 0.134 | 0.190(3) | 0.363(3) | 0.135(2) | 0.14(3) | 0.11(4) | 0.15(3) | –0.02(3) | –0.01(3) | –0.04(3) |
| F(4) | 4*e* | | 0.2282(3) | 0.4446(3) | 0.2236(2) | 0.162(3) | 0.139(3) | 0.067(2) | 0.017(3) | 0.022(2) | –0.002(2) |
| O(1) | 4*e* | | 0.2340(3) | 0.7260(3) | 0.2192(2) | 0.125(3) | 0.121(3) | 0.121(3) | –0.036(3) | 0.071(3) | –0.061(3) |
| P(1) | 4*e* | | 0.51113(6) | 0.68409(8) | 0.19112(5) | 0.0398(5) | 0.0476(6) | 0.0366(5) | –0.0016(4) | –0.0050(4) | 0.0048(5) |
| P(2) | 4*e* | | 0.28504(6) | 0.77814(8) | 0.03165(5) | 0.0393(5) | 0.0396(5) | 0.0376(5) | 0.0009(4) | –0.0028(4) | 0.0017(4) |
| B(1) | 4*e* | | 0.2416(5) | 0.4329(5) | 0.1567(3) | 0.086(4) | 0.044(3) | 0.070(4) | 0.000(3) | 0.007(3) | –0.004(3) |
| C(1) | 4*e* | | 0.5419(3) | 0.8128(3) | 0.2243(2) | 0.042(2) | 0.051(2) | 0.048(2) | 0.001(2) | –0.002(2) | 0.000(2) |
| C(2) | 4*e* | | 0.5355(3) | 0.8976(4) | 0.1800(2) | 0.086(3) | 0.058(3) | 0.059(3) | –0.003(3) | 0.003(2) | 0.004(2) |
| C(3) | 4*e* | | 0.5552(4) | 0.9984(4) | 0.2017(3) | 0.097(4) | 0.054(3) | 0.091(4) | –0.003(3) | 0.009(3) | 0.003(3) |
| C(4) | 4*e* | | 0.5807(4) | 1.0159(4) | 0.2674(3) | 0.095(4) | 0.054(3) | 0.107(4) | 0.004(3) | –0.011(3) | –0.017(3) |
| C(5) | 4*e* | | 0.5853(4) | 0.9354(5) | 0.3126(3) | 0.134(5) | 0.078(4) | 0.078(4) | 0.005(4) | –0.031(3) | –0.024(3) |
| C(6) | 4*e* | | 0.5652(4) | 0.8335(4) | 0.2917(2) | 0.101(4) | 0.058(3) | 0.062(3) | 0.005(3) | –0.022(3) | –0.001(3) |
| C(7) | 4*e* | | 0.6066(3) | 0.6548(3) | 0.1333(2) | 0.047(2) | 0.043(2) | 0.040(2) | 0.004(2) | –0.002(2) | 0.006(2) |
| C(8) | 4*e* | | 0.6991(3) | 0.6987(3) | 0.1384(2) | 0.053(2) | 0.056(3) | 0.051(2) | –0.006(2) | 0.005(2) | 0.001(2) |
| C(9) | 4*e* | | 0.7692(3) | 0.6687(4) | 0.0938(2) | 0.058(3) | 0.069(3) | 0.074(3) | –0.005(2) | 0.013(2) | 0.006(3) |
| C(10) | 4*e* | | 0.7478(3) | 0.5959(4) | 0.0441(2) | 0.074(3) | 0.062(3) | 0.070(3) | 0.008(3) | 0.022(2) | 0.005(3) |
| C(11) | 4*e* | | 0.6575(4) | 0.5518(4) | 0.0382(2) | 0.088(3) | 0.062(3) | 0.066(3) | 0.007(3) | 0.009(3) | –0.012(3) |
| C(12) | 4*e* | | 0.5873(3) | 0.5811(3) | 0.0824(2) | 0.060(2) | 0.060(3) | 0.060(3) | –0.002(2) | –0.002(2) | –0.009(2) |
| C(13) | 4*e* | | 0.5355(3) | 0.5903(3) | 0.2593(2) | 0.051(2) | 0.048(2) | 0.037(2) | –0.004(2) | –0.003(2) | 0.002(2) |
| C(14) | 4*e* | | 0.6290(3) | 0.5602(3) | 0.2800(2) | 0.057(2) | 0.064(3) | 0.051(2) | –0.002(2) | –0.008(2) | 0.011(2) |
| C(15) | 4*e* | | 0.6442(3) | 0.4845(4) | 0.3292(2) | 0.076(3) | 0.068(3) | 0.057(3) | 0.017(3) | –0.010(2) | 0.010(3) |
| C(16) | 4*e* | | 0.5673(4) | 0.4370(4) | 0.3578(2) | 0.109(4) | 0.059(3) | 0.047(2) | 0.014(3) | 0.004(3) | 0.009(2) |
| C(17) | 4*e* | | 0.4753(4) | 0.4656(4) | 0.3378(2) | 0.092(4) | 0.065(3) | 0.062(3) | –0.010(3) | 0.023(3) | 0.013(3) |
| C(18) | 4*e* | | 0.4577(3) | 0.5414(3) | 0.2886(2) | 0.061(3) | 0.059(3) | 0.054(2) | –0.004(2) | 0.008(2) | 0.004(2) |
| C(19) | 4*e* | | 0.1551(2) | 0.7660(3) | 0.0111(2) | 0.041(2) | 0.043(2) | 0.042(2) | –0.002(2) | 0.003(2) | –0.002(2) |
| C(20) | 4*e* | | 0.1109(3) | 0.6710(3) | 0.0202(3) | 0.054(3) | 0.047(3) | 0.110(4) | 0.001(2) | –0.003(2) | 0.009(3) |
| C(21) | 4*e* | | 0.0138(3) | 0.6568(4) | 0.0001(3) | 0.057(3) | 0.056(3) | 0.159(5) | –0.018(2) | 0.000(3) | –0.004(4) |
| C(22) | 4*e* | | –0.0395(3) | 0.7374(4) | –0.0273(2) | 0.044(2) | 0.079(3) | 0.090(3) | –0.004(2) | –0.005(2) | –0.007(3) |
| C(23) | 4*e* | | 0.0014(3) | 0.8323(4) | –0.0336(2) | 0.048(2) | 0.071(3) | 0.066(3) | 0.004(2) | –0.005(2) | 0.014(3) |
| C(24) | 4*e* | | 0.0983(3) | 0.8471(3) | –0.0145(2) | 0.045(2) | 0.050(2) | 0.064(3) | 0.001(2) | –0.006(2) | 0.013(2) |
| C(25) | 4*e* | | 0.3410(3) | 0.7418(3) | –0.0467(2) | 0.047(2) | 0.040(2) | 0.044(2) | 0.002(2) | –0.000(2) | –0.003(2) |
| C(26) | 4*e* | | 0.4404(3) | 0.7238(4) | –0.0457(2) | 0.050(2) | 0.080(3) | 0.063(3) | –0.004(2) | 0.004(2) | –0.018(3) |
| C(27) | 4*e* | | 0.4853(3) | 0.6943(4) | –0.1040(3) | 0.055(3) | 0.093(4) | 0.091(4) | –0.007(3) | 0.021(3) | –0.026(3) |
| C(28) | 4*e* | | 0.4325(4) | 0.6790(4) | –0.1625(2) | 0.081(3) | 0.071(3) | 0.063(3) | –0.007(3) | 0.028(3) | –0.011(3) |
| C(29) | 4*e* | | 0.3349(4) | 0.6959(4) | –0.1651(2) | 0.085(4) | 0.110(4) | 0.049(3) | 0.010(3) | 0.003(2) | –0.018(3) |
| C(30) | 4*e* | | 0.2891(3) | 0.7266(4) | –0.1072(2) | 0.058(3) | 0.089(3) | 0.049(2) | 0.018(2) | –0.002(2) | –0.011(2) |
| C(31) | 4*e* | | 0.3030(2) | 0.9188(3) | 0.0386(2) | 0.040(2) | 0.047(2) | 0.047(2) | –0.002(2) | –0.001(2) | 0.000(2) |
| C(32) | 4*e* | | 0.2851(3) | 0.9660(3) | 0.1000(2) | 0.066(3) | 0.056(3) | 0.060(3) | –0.005(2) | 0.009(2) | –0.009(2) |
| C(33) | 4*e* | | 0.2871(3) | 1.0745(4) | 0.1058(3) | 0.084(3) | 0.059(3) | 0.077(3) | 0.001(3) | 0.007(3) | –0.023(3) |
| C(34) | 4*e* | | 0.3093(4) | 1.1346(4) | 0.0519(3) | 0.087(3) | 0.050(3) | 0.110(4) | –0.007(3) | 0.002(3) | –0.016(3) |
| C(35) | 4*e* | | 0.3297(4) | 1.0893(4) | –0.0084(3) | 0.114(4) | 0.055(3) | 0.091(4) | –0.015(3) | 0.017(3) | 0.010(3) |
| C(36) | 4*e* | | 0.3271(3) | 0.9812(3) | –0.0147(2) | 0.091(3) | 0.050(3) | 0.061(3) | –0.009(2) | 0.014(2) | 0.000(2) |
| C(37) | 4*e* | | 0.1370(4) | 0.6972(5) | 0.2185(3) | 0.090(4) | 0.112(5) | 0.104(4) | 0.014(4) | 0.034(3) | 0.000(4) |

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