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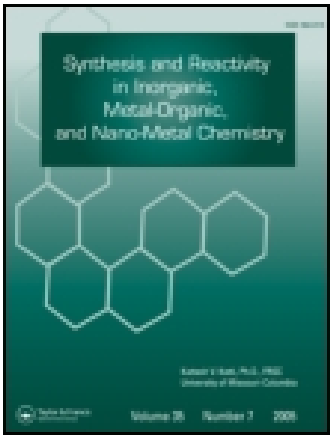

Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry
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Synthesis, Characterization, and Structure of a Silver(I) Compound With Thymine

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Synthesis, Characterization, and Structure of a Silver(I) Compound With Thymine

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The interaction of [Ag(PPh_3)$_2$(CH_3COO)] (1) with thymine led to the displacement of the acetate ligand forming the mononuclear compound [Ag(PPh_3)$_2$($C_5H_5N_2O_2$)] (2). This compound has been characterized by IR, NMR, element analysis, and X-ray crystallographic analysis. Crystal structure of 2 shows each Ag ion is three-coordinated by two P atoms of the triphenylphosphino ligands and one N atoms of the thymine, which is stabilized by two intermolecular N-H. . .O and O-H. . .O hydrogen bonding interactions.

Keywords hydrogen bond, phosphines, silver, thymine

INTRODUCTION

The coordination and supramolecular chemistry of silver(I) compounds received increasing interest due to their antiethylene and antimicrobial activities.[1–6] It is noted that metal complexes of nucleobases are very important in nucleic acid processes and in the development of antitumor therapies.[7–11] Based on these reasons and in continuation of our work on Ag compounds with diphosphine ligand of dppf.[12,13] we wished to synthesize a series of Ag(I) derivatives containing mixed ligands of phosphines and nucleobases, which may have the same interesting activities. Thymine may offer three coordination sites, N1, O1, and O2, of which the amino hydrogen atom is used to be displaced by silver ion in neutral or alkaline solution. Herein, we describe the preparation, characterization, and molecular structure of the phosphinesilver(I) compound with thymine.

EXPERIMENTAL

Materials and Physical Measurements

All reagents and chemicals were purchased commercially and used without further purification, and all water used in experiments was doubly distilled. Infrared (IR) spectra were recorded on a TJ270–30A spectrometer (Tianjin) in KBr pellets in the range of 400–4000 cm^{-1}. ^1H NMR and $^{31}P\{^1H\}$ NMR spectrum were measured in d_6-DMSO on Avance 400 MHz (Bruker) spectrometer. Elemental analyses were carried out on a Perkin-Elmer 2400II elemental analyzer.

The diffraction data were collected on a Bruker APXE II diffractometer equipped with a graphite-monochromatized Mo Kα radiation ($\lambda = 0.71073$ Å) up to a 2θ limit of 54.88° **2** at 296(2) K. Data intensity was corrected by Lorentz-polarization factors and empirical absorption. The structure was solved by direct methods and expanded with difference Fourier techniques. All non-hydrogen atoms were refined anisotropically. Except the hydrogen atoms on oxygen atoms, the other hydrogen atoms were generated geometrically. All calculations were performed with the SHELXTL-97 package. The crystallographic data and structural refinement are summarized in Table 1, the selected bond lengths and bond angles are listed in Table 2.

Synthesis

*[Ag(PPh_3)$_2$ (CH_3COO)] (**1**)*

[Ag(PPh_3)$_2$(CH_3COO)] (**1**) was easily prepared by slightly modifying the literature procedure.[14] PPh_3 (0.9428 g, 3.595 mmol) and AgAc (0.3000 g, 1.797 mmol) in 2:1 molar equivalents were added in mixed solvents of C_2H_5OH -H_2O (30 mL:30 mL), the solution with shielding from light was warmed at 70°C for 5 h gave a transparent liquid. After cooling, evaporation of the solution and washed with diethyl ether to give the colorless residual solids. Yield 1.1111 g, 89%. Anal. Calcd. for $C_{37}H_{33}P_2AgO_2$: C, 66.0; H, 4.8; Found (%): C, 66.1; H, 4.9. ^1H NMR (CD_3OD, ppm): δ 1.90 (s, 3H, CH_3), 7.37 - 7.49 (m, 30H, Ph). $^{31}P\{^1H\}$ NMR (CD_3OD): δ 11.30 (s). IR (KBr, cm^{-1}): ν 3444w, 3062w, ν (CO) 1650w, 1552s, 1480s (Ar), 1435s (Ar), 1400s, 1335m, 1309w, 1186w, 1158w, 1098w, 1072w, 1028w,

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TABLE 1
Selected bond lengths (Å) and angles (°) for **2**

$[Ag(PPh_3)_2]\ C_5H_5N_2O_2 \cdot CH_3OH$	
Bond lengths	
Ag1-P1	2.4513 (10)
Ag1-P2	2.4544 (10)
Ag1-N1	2.2361 (26)
O1-C13A	1.2519 (40)
O2-C5	1.2412 (42)
N1-C13A	1.3369 (43)
N1-C5	1.3525 (44)
N2-C13A	1.3711 (40)
N2-C15A	1.3488 (43)
C19A-C15A	1.3389 (50)
C19A-C5	1.4543 (47)
C19A-C11	1.5069 (49)
Bond angles	
P1-Ag1-N1	117.50 (8)
P1-Ag1-P2	128.33 (4)
P2-Ag1-N1	114.12 (8)
Ag1-N1-C5	119.34 (22)
Ag1-N1-C13A	118.47 (21)
C13A -N1-C5	112.16 (27)
N1-C5-O2	118.67 (31)
N1-C5-C19A	119.00 (31)
O2-C5-C19A	112.33 (33)
C5-C19A-C11	119.83 (33)
C5-C19A-C15A	116.74 (30)
C15A-C19A-C11	123.34 (32)
C19A- C15A -N2	112.15 (29)
C15A -N2-C13A	121.24 (29)
N2-C13A-O1	119.47 (30)
N2-C13A-N1	118.70 (29)
O1-C13A-N1	121.84 (28)

998m, 918w, 768m, 745s, 696s, 658m, 618w, 515s, 501s, 440w, 419w.

*$[Ag(PPh_3)_2(C_5H_5N_2O_2)]$ (**2**)*

A mixture of **1** (0.0691 g, 0.100 mmol) and thymine (0.0126 g, 0.100 mmol) in 1:1 molar ratio was dissolved in mixed solvents of CH_3OH-H_2O (10 mL:10 mL), and to this solution was added a solution of sodium hydroxide to adjust the pH between 7 and 8. The resulting mixture was heated at 70°C for 2 h. After cooling, the resultant suspension was filtered to collect an insoluble colorless precipitate, which was washed with diethyl ether and dried *in vacuo*. Yield 0.0305 g (37%). Anal. Calcd. for $C_{42}H_{39}N_2P_2O_3Ag$: C, 63.8; H, 5.0; N,3.5; Found (%):C, 63.7; H, 5.1; N, 3.5. ^1H NMR (d_6-DMSO, ppm): δ 1.69 (s, 3H, CH_3), 3.16 (s, 1H, CH), 7.33–7.40 (m, 25H, Ph), 7.45–7.48 (m, 5H, Ph). $^{31}P\{^1H\}$ NMR (d_6-DMSO): δ 7.19 (s). IR (KBr, cm^{-1}): 3444w, ν (NH) 3058w, ν (CO) 1638s, ν (C=N) 1547s, ν (NH) 1494m, ν (C=N) 1485s, 1434s, 1414s, 1360w, 1294w, ν (C-N) 1181w, 1098m, 1030w, 1000w, 890w, 787m, 744s, 696s, 589w, ν (Ag-N(ligand)) 509s.

Diffraction-quality crystals of **2**·MeOH was obtained by slow crystallization of the complex from CH_3OH-H_2O after five days at room temperature.

RESULTS AND DISCUSSION

Synthesis

Compound **1** is readily obtained in high yield by reaction of $Ag(CH_3COO)$ and PPh_3 in 1:2 molar ratio. Quality diffraction crystals of **1** was unexpectedly obtained from recrystallization of complex **2** in MeOH/diethyl ether, its structural data is consistent with the values reported by Ng and Othman in 1997.[14] Unfortunately, their report does not include the spectral properties. In the IR spectrum, the characteristic band at 1650 cm^{-1} is assigned to ν(C=O) group of acetate ligands. The ^1H NMR spectrum displays an intense singlet at δ 1.9 ppm and a multiplet at δ 7.37–7.49 ppm due to –CH_3 of acetate and phenyl protons of PPh_3, respectively. $^{31}P\{^1H\}$ NMR spectrum shows only one resonance at δ 11.3 ppm, consistent with its solid-state structure.

Repeated attempts to prepare compound **2** by treating compound **1** and thymine in hydrothermal method were largely unsuccessful and gave black solids, which are insoluble in the polar and chlorinated solvents. However, the equimolar reaction between **1** and thymine in CH_3OH-H_2O solvents yielded compound **2** as white precipitates. In this reaction, it is necessary to add a base for this reaction to remove the produced CH_3COOH, indicating that the product toward hydrolysis with CH_3COOH is not robust. At elevated pH, the amino hydrogen atom of thymine is deprotonated and coordinated with Ag atom, instead of displacing PPh_3 as ligand to form silver complex containing both thymine ligand and acetate anion. Because silver is a hard atom and as such would be expected to react with nitrogen donor over an oxygen atom, and thus are available for acetate

TABLE 2
Hydrogen bonds for **2** (Å and °)

D-H	d(D-H)	d(H..A)	<DHA	d(D..A)	A	symmetry codes
O3-H3	0.835	1.881	174.52	2.713	O2	
N2-H2B	0.860	1.932	166.88	2.776	O1	[-x+1, -y+1, -z+1]

ligand replacement in compound **1**, resulting in the formation of **2**. Compound **2** is soluble in methanol and ethanol, and is stable and shows no sensitivity to the light as solids and in solution at room temperature. IR spectrum of **2** shows the characteristic bands at 3058 and 1494 cm^{-1} are due to ν(N-H) stretching and bending vibration. Two strong bands at 1547 and 1485 cm^{-1} are assigned to ν(C=N) stretching frequencies. A strong and sharp band at 1638 cm^{-1} is related to the ν(C=O) stretching vibration for the carbonyl groups, which is shifted about 38 cm^{-1} toward lower wave numbers in caparison to the free thymine ligands (1676 cm^{-1}). The ^{1}H NMR spectrum displays signals of the methyl group CH_3 and CH as a singlet, respectively, and the phenyl protons of PPh_3 ligands as two multiplets. We note but cannot explain the absence of the NH proton. The $^{31}P\{^1H\}$ NMR spectrum gives only one relatively sharp signal at δ 7.19 ppm, shifted to the low field as compared with δ 11.3 ppm in complex **1**, and may be due to that oxygen atom is more electronegative than that of nitrogen atom. Solid-state emission spectra of free thymine, and compounds **1** and **2** have been investigated at room temperature as well, however, the results exhibit that these two silver(I) complexes unfortunately emit no photoluminescence, in agreement with those reported Ag(I) complexes with weak photoluminescence.[15]

X-Ray Crystal Structure

*$[Ag(PPh_3)_2(C_5H_5N_2O_2)]$ (**2**)·CH_3OH*

The molecular structure of compound **2** is shown in Figure 1, compound **2** is mononuclear, crystallizes in the P-1 space group, in which the silver atom is three-coordinated with two phosphines and one thymine ligand and exhibits 16e trigonal planar geometry because of the lack of electron donor. The distances of Ag-P (Ag–P1 2.4513 (10) Å, Ag–P2 2.4544 (10) Å) are not different from the reported Ag(PPh_3) complexes (e.g., 2.436 (3) Å, 2.432 (2) Å in [Ag(R,S-Hpyrrld)$(PPh_3)_2$],[16] 2.6026 (8) Å, 2.5441 (7) Å, and 2.5432 (7) Å in $AgOCOC_2F_5\cdot PPh_3\cdot 0.5thf$).[17] However, the Ag-N distance (2.2361 (26) Å) is significantly longer than those reported Ag(I) complexes with nucleobases ($AgNO_3\cdot$9-methyladenine, 2.08–2.11 Å[18]; $Ag(C_5H_5N_2O_2)$, 2.08(3) Å).[19] The bond lengths of C=O and C=C double bond are 1.2519(40), 1.2412(42), and 1.3389(50) Å, respectively. Another feature of interest is the bond length of single-bond character present in C13A-N1 bond, which exhibits partial double-bond character (1.3369(43) Å), close to those of $[CpRu(dppf)(S_2CNEt_2)_2]$.[20] The sum of angles at Ag is 359.95° (P1- Ag1 – N1, 117.50(8)°; P1- Ag1 – P2, 128.33(4)°; P2- Ag1 – N1, 114.12(8)°), indicating that Ag, P1, P2, and N3 are coplanar. The same coplanar cases were found in the ring of thymine, as the sum of angles at C13A and C19A are 360.01° (N1-C13A-O1 121.84(28)°, O1-C13A-N2 119.47(30)°, N1-C13A-N2 118.70(29)°), and 359.91° (C5-C19A-C11 119.83(33)°, C5-C19A-C15A 116.74(30)°, C11-C19A-C15A 123.34(32)°), respectively. Moreover, the conformation stabilized by secondary bonding interactions were observed as shown in Figure 2. The hydrogen bonds data are summarized in Table 3. The intermolecular hydrogen bonds exist between N-H of one thymine and O atom of another thymine with N…O and O…H distances of 2.713 and 1.881 Å and an N-H…O angle of 166.88°, another intermolecular hydrogen bond O-H…O occurs between methanol solvates and thymine, with O…O and O…H distances of 2.776 and 1.932 Å and an O-H…O angle of 174.52°, in which this interaction must be considered very strong (sum of the covalent

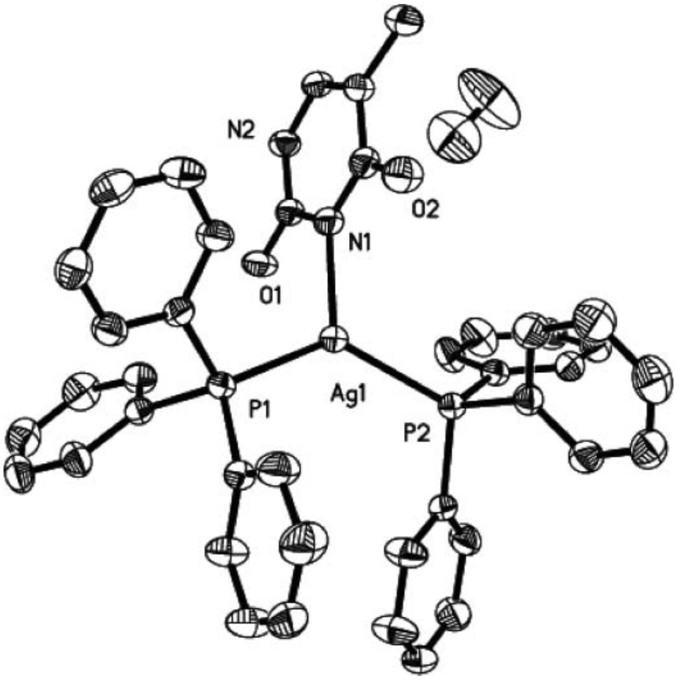

FIG. 1. ORTEP's molecular structure of **2**.

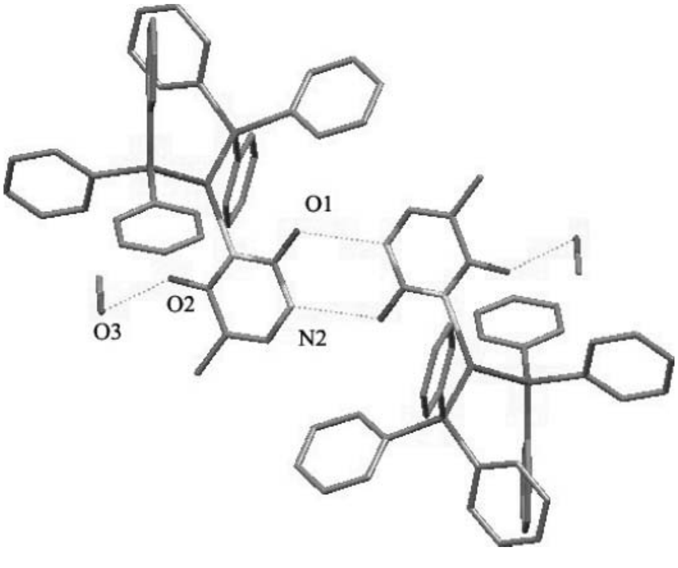

FIG. 2. Hydrogen bonding interactions in **2**.

TABLE 3
Crystal data and structure refinement for **2**

Compound	$[Ag(PPh_3)_2C_5H_5N_2O_2]\cdot CH_3OH$
Empirical formula	$C_{42}H_{39}N_2P_2AgO_3$
Formula weight	789.5944
Temperature (K)	296(2) K
Crystal system	Triclinic
Space group	P-1
a (Å)	10.454(2)
b (Å)	10.494(2)
c (Å)	17.580(4)
α (°)	98.73(3)
β (°)	91.39(3)
γ (°)	95.16(3)
V (Å^3)	1897.3(7)
Z	2
Dc(g·cm^{-1})	1.382
μ(mm^{-1})	0.656
F(000)	812
Reflections collected	18186 / 8480
Independent reflections	8480 [R(int) = 0.0259]
Completeness to theta = 27.53	97.7%
Data / restraints / parameters	8480 / 1 / 455
goodness-of-fit on F^2	1.140
Final R indices[$I>2\sigma(I)$]	R = 0.0395, wR = 0.1174
R indices (all data)	R = 0.0509, wR = 0.1304
Largest diff. peak and hole	1.075,-0.413

van der Waals radii = 3.04 Å). However, these intermolecular hydrogen bondings do not form a supramolecular network.

SUPPLEMENTARY MATERIALS

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre; CCDC No. 814234 for **2**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB121EZ, UK (Fax: +44–1223–336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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