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Synthesis, spectroscopic and X-ray structural studies of silver(I) complexes of α -keto stabilized phosphorus ylides

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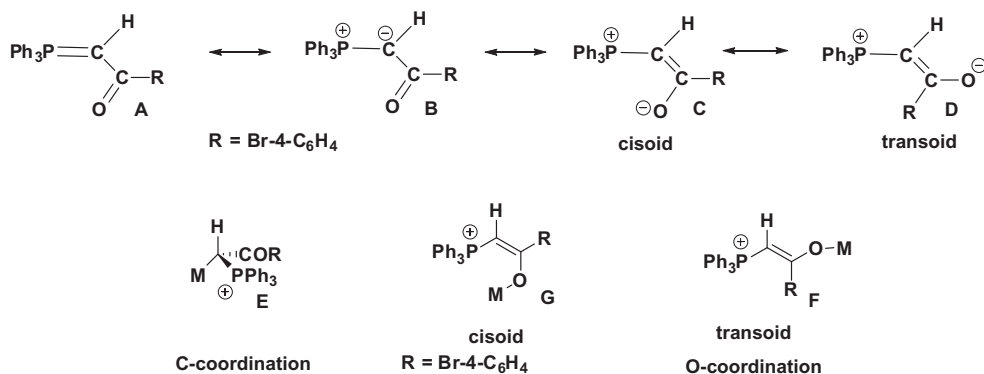
Reactions between AgX (X = OTf, NO₃) and ylides of the type Ph₃P = CHC(O)C₆H₄-4-R (R = F, Br, OMe) in molar ratio 1 : 2 lead to complexes [Ag{CH(PPH₃)C(O)C₆H₄-4-R}]X (**1–4**); X-ray structure determinations have been carried out on **4**. The IR and NMR data of the products formed by reaction of Ag(I) with the ylides are consistent with C-bounded ylides. Analytical data indicate 1:2 stoichiometry between the ylide and Ag(I) in the products. The molar conductivities of these complexes are within the range for 1:1 electrolytes. Crystallographic data for **4** are: crystal system, triclinic; space group, *P* $\bar{1}$, *a* = 12.1151(4), *b* = 13.8989(5), *c* = 15.4855(5) Å, β = 102.676(3)°, *V* = 2477.82(15) Å³, *Z* = 2.

Keywords: Phosphorus ylide; Silver(I) complexes; X-ray crystal structure

1. Introduction

The utility of metalated phosphorus ylides in synthetic chemistry has been well documented [1, 2]. Synthesis of complexes derived from ylides and Ag(I) began in 1975 by Yamamoto *et al.* [3]; other types of ylide complexes of silver(I) have been reported in [4–9]. In 1987 and 1993, Vicente *et al.* [10, 11] reported crystal structures of Ag(I) complex of phosphorus ylides. The α -keto-stabilized phosphorus ylides show interesting properties such as high stability and ambidentate character as ligands (C- vs. O-coordination) [12]. This ambidentate character can be rationalized in terms of resonance forms A–C, together with the isomeric form D (scheme 1). Form B leads to coordination by the carbon to give a complex of form E, whereas isomers C and D would both lead to coordination by oxygen, affording structures F (transoid) and G (cisoid), respectively. Although many coordination modes are possible for keto ylides [13], coordination through carbon is dominant, especially with soft metal ions, e.g. Pd(II), Pt(II), Ag(I), Hg(II), Au(I), and Au(III) [14–17], and very few examples of O-coordinated ylides are known [8]. Some of these contain the ylide O-coordinated to a hard metal, such as Sn(IV) [18] or a group with a high oxidation number [19].

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Scheme 1.

Only W(0) complexes of the type W(CO)₅L (L = ylide) [20] and some Pd(II) complexes contain stable ylides O-linked to a soft metal center [21]. In this study, we describe the preparation and spectroscopic characterization (IR and NMR) of Ag(I) complexes with the title ylides. The data collected and single crystal X-ray diffraction of **4** demonstrates the C-coordination of the ylide to the metal.

2. Experimental

2.1. Materials

Silver triflate and silver nitrate, 2-bromo-4'-fluoroacetophenone, 2-bromo-4'-chloroacetophenone, 2,4'-di-bromoacetophenone, 2-bromo-4'-methoxyacetophenone, and triphenylphosphine were purchased from Merck. The ylides were synthesized by reaction of triphenylphosphine with a chloroform solution of 2-bromo compounds and deprotonated by NaOH [12]. All solvents were dried by reported methods [22].

2.2. Physical measurements

All solvents were of reagent grade and used without purification. Solution-state ¹H- and ³¹P-NMR spectra at 300 K were obtained in CDCl₃ using a 500 MHz Bruker spectrometer operating at 500.13 MHz for ¹H and 161.97 MHz for ³¹P, referenced to H₃PO₄ (85%) for ³¹P{¹H}-NMR spectra. IR spectra were recorded on a FT-IR JASCO 680 spectrophotometer as KBr disks. Melting points were measured on a Gallenamp 9B 3707 F apparatus.

2.3. Synthesis of the complexes

2.3.1. [Ag(FBPPY)₂NO₃ (1). The ylide FBPPY (0.470 g, 1.18 mmol, see table 1 for abbreviations) was added to a solution of AgNO₃ (0.100 g, 0.59 mmol) in

Table 1. $\nu(\text{CO})$ and $\nu(\text{P-C})$ (cm^{-1}) of selected phosphoranones and their metal complexes.

Compound	$\nu(\text{CO})$	$\Delta(\text{CO})$	$\nu(\text{P-C})$	Reference
$\text{Ph}_3\text{PCHCOPh}$ (BPPY)	1520	–	878	[18]
$\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{-Br}$ (=BrBPPY)	1518	–	878	This work
$\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{-Cl}$ (=CBPPY)	1521	–	881	This work
$\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{-F}$ (=FBPPY)	1516	–	883	This work
$\text{Ph}_3\text{PCHCOC}_6\text{H}_4\text{-OMe}$ (=MOBPPY)	1504	–	855	This work
$\text{Ag}(\text{FBPPY})_2\text{NO}_3$ (1)	1613	+97	880	This work
$\text{Ag}(\text{CBPPY})_2\text{NO}_3$ (2)	1616	+95	875	This work
$[\text{Ag}(\text{Br-BPPY})_2]\text{CF}_3\text{SO}_3$ (3)	1615	+97	872	This work
$[\text{Ag}(\text{MOBPPY})_2]\text{CF}_3\text{SO}_3$ (4)	1612	+108	844	This work

acetone (10 mL). The solution was stirred for 1 h during which it was protected from light and then filtered. The volume of solvent was reduced under vacuum to 2 mL. Diethyl ether (25 mL) was added to precipitate $[\text{Ag}\{\text{CH}(\text{PPh}_3)\text{C}(\text{O})\text{C}_6\text{H}_4\text{-4-F}\}]\text{NO}_3$ (**1**) as a white powder. Yield: 65%; m.p.: 185°C (dec). Anal. Calcd for $\text{C}_{52}\text{H}_{40}\text{AgF}_2\text{NO}_5\text{P}_2$: C, 64.60; H, 4.17; N, 1.45. Anal. Found: C, 64.54; H, 4.21; N, 1.34. $^1\text{H-NMR}$: (500 MHz, CDCl_3 , RT): δ (ppm): 5.06 (br s, 1H), 7.1–8.1 (m, 19H, Ph) ppm and $^{31}\text{P-NMR}$: 22.13 ppm, $\Lambda = 122 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$.

2.3.2. $[\text{Ag}(\text{CBPPY})_2]\text{NO}_3$ (2**).** The ylide CBPPY (0.489 g, 1.18 mmol) was added to a solution of AgNO_3 (0.100 g, 0.59 mmol) in acetone (10 mL). The solution was stirred for 1 h during which it was protected from light and then filtered. The volume of solvent was reduced under vacuum to 2 mL. Diethyl ether (25 mL) was added to precipitate $[\text{Ag}\{\text{CH}(\text{PPh}_3)\text{C}(\text{O})\text{C}_6\text{H}_4\text{Cl}\}]\text{NO}_3$ (**2**) as a white powder. Yield: 81%; m.p.: 179°C. Anal. Calcd for $\text{C}_{52}\text{H}_{40}\text{AgCl}_2\text{NO}_5\text{P}_2$: C, 62.47; H, 4.03; N, 1.4. Anal. Found: C, 62.54; H, 3.92; N, 1.58. $^1\text{H-NMR}$: (500 MHz, CDCl_3 , RT): δ (ppm): 6.06 (br s, 1H, CH), 7.2–8.2 (m, 19H, Ph) ppm and $^{31}\text{P-NMR}$: 21.79 ppm, $\Lambda = 137 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$.

2.3.3. $[\text{Ag}(\text{Br-BPPY})_2]\text{CF}_3\text{SO}_3$ (3**).** BrBPPY (0.918 g, 2 mmol) was added to a solution of AgOTf (0.256 g, 1 mmol) in acetone (10 mL). The solution was stirred for 1 h during which it was protected from light and then filtered. The volume was reduced under vacuum to 2 mL and *n*-hexane (25 mL) was added to precipitate $[\text{Ag}\{\text{CH}(\text{PPh}_3)\text{C}(\text{O})\text{C}_6\text{H}_4\text{-4-Br}\}_2]\text{OTf}$ (**3**) as a white powder. Yield: 71%; m.p.: 161°C; $^1\text{H-NMR}$: (500 MHz, CDCl_3 , RT): δ (ppm) 8.06 (d, 2H, 2H, C_6H_4 , $^3J_{\text{H-H}} = 8.5$ Hz), 7.56–7.30 (m, 15H, $3\text{C}_6\text{H}_5$), 6.81 (d, 2H, 2Hm, C_6H_4 , $^3J_{\text{H-H}} = 8.8$ Hz), 5.22 (br s, 1H, CHP), 3.86 (s, 3H, OMe); $^{31}\text{P-NMR}$: (500 MHz, CDCl_3 , RT): δ (ppm) 24.75 (s, PPh_3). Anal. Calcd for $\text{C}_{55}\text{H}_{46}\text{Ag-F}_3\text{O}_7\text{P}_2\text{S}$: C, 61.3; H, 4.3; S, 3.0. Anal. Found: C, 61.5; H, 3.9; S, 3.1; $\Lambda = 131 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$.

2.3.4. $[\text{Ag}(\text{MOBPPY})_2]\text{CF}_3\text{SO}_3$ (4**).** The MOBPPY (0.410 g, 1 mmol) was added to a solution of AgOTf (0.256 g, 1 mmol) in acetone (10 mL). The solution was stirred for 1 h during which it was protected from light and then filtered. The volume was reduced under vacuum to 2 mL and diethyl ether (25 mL) added to precipitate $[\text{Ag}\{\text{CH}(\text{PPh}_3)\text{C}(\text{O})\text{C}_6\text{H}_4\text{-OMe}\}_2]\text{OTf}$ (**5**) as a white powder. Yield: 58%; m.p.: 176°C;

$^1\text{H-NMR}$: (500 MHz, CDCl_3 , RT): δ (ppm) 8.06 (d, 2H, 2Ho, C_6H_4 , $^3J_{\text{H-H}} = 8.5$ Hz), 7.56–7.30 (m, 15H, $3\text{C}_6\text{H}_5$), 6.81 (d, 2H, 2Hm, C_6H_4 , $^3J_{\text{H-H}} = 8.8$ Hz), 5.22 (br s, 1H, CHP), 3.86 (s, 3H, OMe); $^{31}\text{P-NMR}$ (500 MHz, CDCl_3 , RT): δ (ppm) 24.75 (s, PPh_3). Anal. Calcd for $\text{C}_{55}\text{H}_{46}\text{AgF}_3\text{O}_7\text{P}_2\text{S}$: C, 61.3; H, 4.3; S, 3.0. Anal. Found: C, 61.5; H, 3.9; S, 3.1; $\Lambda = 72 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$ [23].

3. Results and discussion

3.1. Synthesis

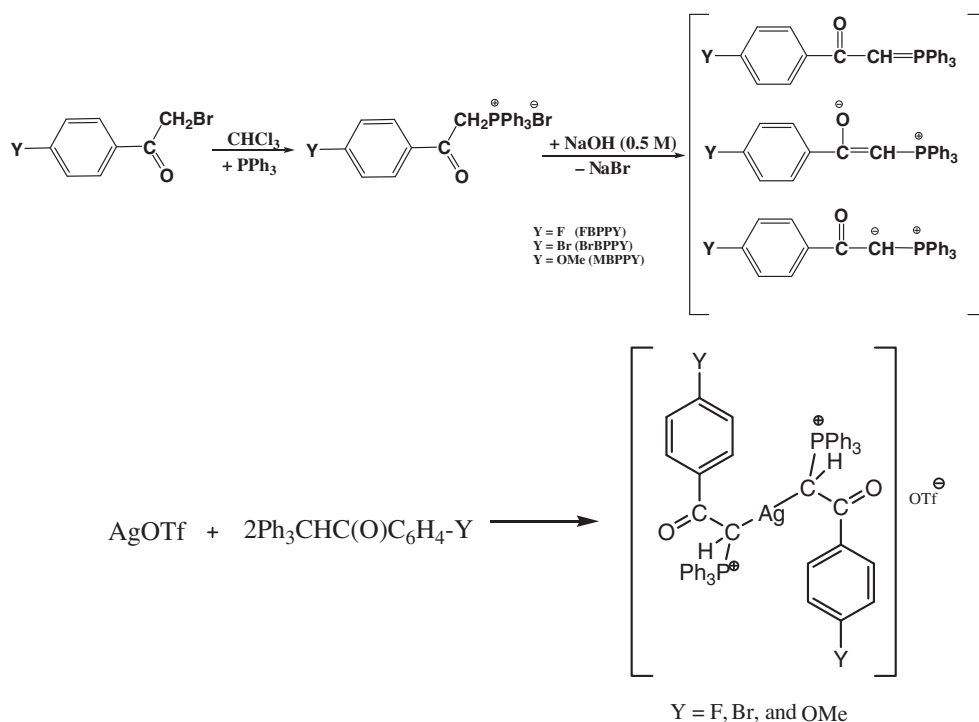
The ligands were synthesized by treating 2-bromo-compound with triphenylphosphine and removal of the proton from the phosphonium salt. Reactions of AgX ($\text{X} = \text{CF}_3\text{SO}_3$ or NO_3) with the ylides in a 1:2 stoichiometry afforded the C-coordinated complexes with a *bis* ylide structure (scheme 2).

3.2. Infrared spectra

The $\nu(\text{CO})$ values, which are sensitive to complexation, occur from 1504 to 1521 cm^{-1} in the parent ylides [18] increases for all complexes (table 1), indicative of coordination of the ylide through carbon. The $\nu(\text{P}^+-\text{C}^-)$, which is also diagnostic for coordination, occurs at 897 cm^{-1} in $\text{Ph}_3\text{P}^+-\text{CH}_2^-$ and at 883 , 881 , 878 , and 855 cm^{-1} in FBPPY, CBPPY, BrBPPY and MOBPPY, respectively, shifted to lower frequencies for complexes suggesting removal of electron density in the P–C bond.

3.3. NMR spectral data

The $^1\text{H-NMR}$ data for the Ag(I) complexes and parent ylides are listed in table 2. The methine proton was broad for **1**, **2**, **3** and **4**, indicating that the ylide dissociates in solution. Compounds wherein the ylide is C-coordinated exhibit a $^2J_{(\text{PH})}$ value of 10 Hz or less [11, 21]. The $^{31}\text{P-NMR}$ resonances of the complexes occur at lower field with respect to free ylide (table 2). The expected downfield shifts of ^{31}P and ^1H signals for the PCH group upon complexation were observed. The appearance of single signals for the PCH group in both the ^{31}P and ^1H spectra at ambient temperature indicates the presence of only one molecule for all the complexes as expected for C-coordination; O-coordination of the ylide sometimes leads to formation of *cis*- and *trans*-isomers giving rise to two different signals in the ^{31}P - and $^1\text{H-NMR}$ [14]. Although two diastereoisomers (RR/SS and RS) are possible for each complex (because the methine carbons are chiral) NMR spectroscopy does not distinguish them at room temperature. Methine resonances are intermediate between, and $^2J_{(\text{PH})}$ values smaller than, those in the free ylides and phosphonium salts as observed for other C-coordinated carbonyl-stabilized phosphorus ylide complexes, due to hybridization change in the ylidic carbon (sp^2 – sp^3) with C coordination [6, 11, 14]. Much larger values of $^2J_{(\text{PH})}$ (*ca* 20 Hz) have been observed in complexes where coordination is through the oxygen [21]. Neither H–Ag nor P–Ag coupling was observed at room temperature in the spectra [16],



Scheme 2.

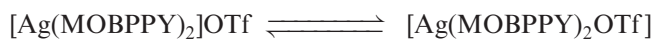
Table 2. ^1H - and ^{31}P -NMR data of phosphorus ylides and complexes with Ag(I).

Compound	^1H Chemical shifts (CH) (δ ppm)	$^2J_{(\text{PH})}$ (Hz)	^{31}P Chemical shifts (δ ppm)
BrBPPY	4.2 (d)	24.02	16.5
FBPPY	4.37 (d)	23.99	16.8
CBPPY	4.4 (d)	23.87	16.7
MOBPPY	4.37 (d)	24.12	16.8
1	5.06 (br s)	—	22.13
2	6.06 (br s)	—	21.79
3	5.32 (br s)	—	22.62
4	5.22 (br s)	—	24.75

possibly a fast equilibrium between complexes and free ylides is responsible for the failure to observe NMR couplings or presence of two diastereoisomers.

3.4. Conductometry measurements

The molar conductivities of **1–3** are within the range for 1:1 electrolytes [24], while the low value found for **4** can be explained by assuming that the anion interacts with the cation in solution [11]:



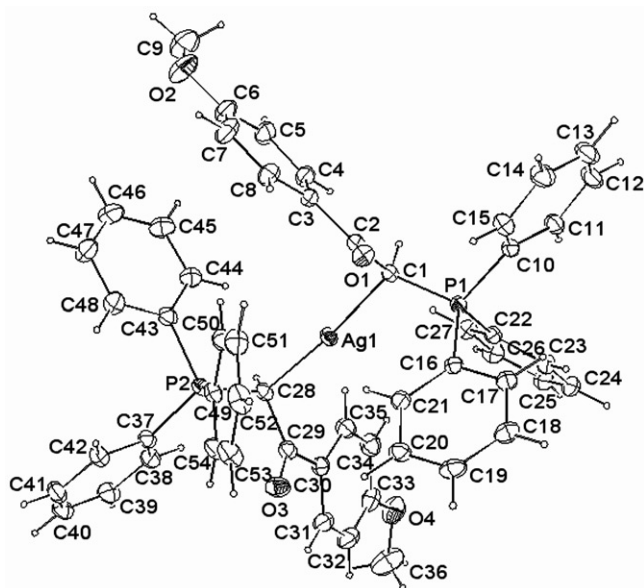


Figure 1. ORTEP plot of **4** (the $\text{CF}_3\text{O}_3\text{S}$ moiety is not shown for clarity).

3.5. Crystal structure

Crystals of **4** were obtained by slow diffusion of *n*-hexane into a CH_2Cl_2 solution at room temperature. The complex is shown in figure 1, crystallographic details in table 3 and significant bond distances and angles are collected in table 4. The Ag(I) atom is located in a slightly distorted linear environment, surrounded by C atoms of the methine of the ylide.

The relative configuration at the chiral carbon atoms are RR. The conformation about the molecular axis (C(1) \cdots C(28)) is almost eclipsed (figure 1). The coordination geometry at silver is essentially linear, although bond lengths and angles at the silver are slightly distorted (C(1)–Ag–C(28) 175.17 (11)). The P(1)–C(1) (methine) distance is 1.758(3) Å, shorter than the average P–C(Ph) single bond distance (1.807 Å), indicating some multiple bond character ($p \cdots c$) [25]. For comparison, the P–C bond distance in Ph_3PCH_2 is 1.66 Å, which corresponds to a bond order of 1.33 [18, 26, 27]. The C(1)–C(2) distance is 1.461(5) (4) Å, indicating single bond character. The C(2)–O(1) distance is 1.233(4) Å, similar to C–O bond distances found in C-bound ylides [28–30]. Complex **4** is an example of an ylide C-bound to a soft metal center.

4. Conclusions

We describe the synthesis and characterization of mononuclear Ag(I) complexes of phosphorus ylides. On the basis of the physico-chemical and spectroscopic data, we propose monodentate C-coordination to the metal, which is further confirmed by the X-ray crystal structure of **4**.

Table 3. Crystal data and structure refinement for **4**.

Empirical formula	C ₅₅ H ₄₆ AgF ₃ O ₇ P ₂ S
Formula weight	1077.80
Temperature (K)	296
Radiation, wavelength	Mo-K α , 0.71073
Measurement device type	Stoe IPDSII
Crystal system, space group	Triclinic, $P\bar{1}$
Unit cell dimensions (\AA , $^\circ$)	
<i>a</i>	12.1151(4)
<i>b</i>	13.8989(5)
<i>c</i>	15.4855(5)
α	96.874(3)
β	102.676(3)
γ	99.292(3)
<i>V</i> (\AA^3)	2477.82(15)
<i>Z</i>	2
<i>D</i> _{Calcd} (g cm ⁻³)	1.445
Crystal color and size (mm ³)	White, 0.71 × 0.34 × 0.07
μ (mm ⁻¹)	0.578
<i>F</i> (000)	1104
θ Range ($^\circ$)	1.5–26.5
Index range	–15 ≤ <i>h</i> ≤ 15, –17 ≤ <i>k</i> ≤ 17, –19 ≤ <i>l</i> ≤ 19
Reflection collected	35,940
Independent reflections	10,274 [<i>R</i> _{int} = 0.0409]
Reflections with $F^2 > 2\sigma$	8172
Absorption correction	Integration (X-RED32, 2002)
Max. and min. transmission	0.9660 and 0.8025
Data/restraints/parameters	10274/0/622
Goodness-of-fit on F^2	1.045
Final <i>R</i> indices [$F^2 > \sigma(F^2)$]	<i>R</i> ₁ = 0.0600, <i>wR</i> ₂ = 0.1108
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0431, <i>wR</i> ₂ = 0.1042

Table 4. Selected bond lengths (\AA) and angles ($^\circ$) for **4**.

Ag1–C1	2.183(3)	O1–C2	1.233(4)	P2–C43	1.810(3)
Ag1–C28	2.175(3)	O3–C29	1.227(4)	C2–C3	1.496(4)
P1–C1	1.758(3)	P1–C10	1.810(3)	C29–C30	1.498(5)
P1–C28	1.761(4)	C1–C2	1.461(5)	C28–C29	1.471(4)
Ag1–C1–P1	114.46(14)	C1–Ag1–C28	175.17(11)	C28–P2–C37	112.08(15)
Ag1–C1–C2	104.4(2)	C1–C2–C3	119.5(3)	C37–P2–C49	109.54(16)
Ag1–C28–P2	105.50(14)	C28–C29–C30	118.8(3)	O1–C2–C1	120.9(3)
Ag1–C28–C29	107.5(2)	P1–C1–C2	114.6(2)	O3–C29–C28	121.2(3)
C1–P1–C10	112.21(14)	P2–C28–C29	112.8(3)		

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