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Pyridine-2-thione (pySH) derivatives of silver(I): Synthesis and crystal structures of dinuclear [Ag₂Cl₂(μ-S-pySH)₂(PPh₃)₂] and [Ag₂Br₂(μ-S-pySH)₂(PPh₃)₂] complexes

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Abstract

Reaction of silver(I) halides with PPh₃ in acetonitrile and then with pyridine-2-thione (pySH) chloroform (1:1:1 molar ratio) has yielded sulfur bridged dimers of general formula, $[Ag_2X_2(\mu-S-pySH)_2(PPh_3)_2]$ (X = Cl, 1, Br, 2). Both these complexes have been characterized using analytical data, NMR spectroscopy and single crystal X-crystallography. The central Ag₂S₂ cores form parallelograms with unequal Ag–S bond distances (2.5832(8), 2.7208(11) Å) in 1 and (2.6306(4), 2.6950(7) Å) in 2, respectively. The Ag···Ag contacts of compounds 1 and 2 are 3.8425(8) and 3.8211(4) Å, respectively. The angles around Ag (in the range 87.19(2)–121.71(2)° in 1 and 87.81(2)–121.53(2)° in 2) reveal highly distorted tetrahedral geometry. There are inter dimer π – π stacking interactions between pyridyl rings (inter ring distances of 3.498 and 3.510 Å in complexes 1 and 2, respectively). The solution state ³¹P NMR spectroscopy has shown the existence of both monomers and dimers. The studies reveal relatively weaker intramolecular –NH···Cl hydrogen bonding in case of AgCl vis-à-vis that in CuCl which favored both a monomer and a dimer with AgCl, and only a monomer with CuCl. © 2008 Elsevier Ltd. All rights reserved.

Keywords: Silver(I); Pyridine-2-thione; Triphenyl phosphine; Crystal structure

1. Introduction

Pyridine-2-thione (I, pySH) bearing the functional group, $-N(H)-C(=S)-\leftrightarrow -N=C(-SH)-$, is the simplest prototype of heterocyclic thioamides and it has shown diverse bonding properties both in neutral and in anion forms (after losing -NH hydrogen) and has formed monomers, dimers, oligomers and polymers [1–8]. Among the coinage metals, neutral pyridine-2-thiones have shown modes IIA–IIC with copper(I) halides [1–8]. As regards silver(I), mode IIA has been shown by py2SH/allied heterocyclic thioamides in distorted tetrahedral complexes, [Ag(py2SH)(PPh_3)_2Cl] [9a], [Ag(pySH)_2(PPh_3)_2](NO_3) [9b], [Ag(pySH-1-Me)_4](BF_4) [9c], [AgL_2(PPh_3)_2](NO_3) (L = benzoxazoline-2-thione) [9d], in trigonal planar com-

plexes, $[AgL_2(SCN)]$ (L = 1, 3-imidazolidine-2-thione) [9e] and $[Ag(pymSH)(PPh_3)_2](NO_3)$ (pymSH = pyrimidine-2thione) [9b] and in a linear complex, $[AgL_2](ClO_4)$ (L = 1, 3-imidazolidine-2-thione) [9f]; while mode IIB was shown in dimers, $[Ag_2Br_2(pymSH)_2(PPh_3)_2]$ [10a], $[Ag_2(\mu-S-pySH)_2(\mu-P,P-dppb)_2](NO_3)_2$ (dppb = Ph_2P-(CH_2)_4-PPh_2) [10b], in polymers {Ag(pySH)Cl}_n [10c] and [{ $Ag(pySH)_2$ }(ClO₄)(H₂O)_{0.5}]_n [9f]; Mode IID with a μ_4 -S mode of bonding of neutral pySH has been shown in { $Ag_4(pySH)_6(NO_3)_4$ }_n polymer [11].



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While several dimeric complexes of copper(I) halides with heterocyclic thioamides are reported [12], there are only two dimeric complexes of heterocyclic thioamides with silver(I), namely, $[Ag_2Br_2(pymSH)_2(PPh_3)_2]$ (pymSH = pyrimidine-2-thione) [10a] and $[Ag_2(\mu-S-pySH)_2-(\mu-P,P-dppb)_2](NO_3)_2$ (dppb = Ph₂P-(CH₂)₄-PPh₂) [10b]. In this paper, dimeric complexes of silver(I) halides with pyridine-2-thione are reported and characterized using analytical data, NMR spectroscopy and single crystal X-ray crystallography.

2. Experimental

2.1. Materials and techniques

Pyridine-2-thione and triphenyl phosphine were procured from Aldrich–Sigma Ltd. Silver(I) halides were freshly prepared from AgNO₃ and NaBr, or KCl and dried *in vacuo*. Elemental analysis for C, H and N were carried out using a thermoelectron FLASHEA1112 analyser. The melting points were determined with a Gallenkamp electrically heated apparatus. The IR spectra of the ligands and the complexes were recorded on the FTIR-SHIMADZU 8400 Fourier Transform spectrophotometer in the range, 4000–500 cm⁻¹ (using KBr pellets). ¹H NMR spectra were recorded on a JEOL AL300 FT spectrometer at 300 MHz in CDCl₃ with TMS as the internal reference. The ³¹P NMR spectra were recorded at 121.5 MHz with (CH₃O)₃P as the external reference taken as zero position.

2.2. Synthesis of $[Ag_2(\mu-S-pySH)_2(PPh_3)_2Cl_2]$ (1)

To AgCl (0.025 g, 0.17 mmol) suspended in 20 mL of acetonitrile was added solid PPh₃ (0.045 g, 0.17 mmol) and stirring was carried out for 24 h. Acetonitrile was removed from the white colored precipitates and chloroform (15 mL) was added. To these precipitates suspended in chloroform, solid pyridine-2-thione (0.019 g, 0.17 mmol) was added, and the contents stirred for 1 h until clear solution was obtained. Slow evaporation of the solution at room temperature formed a solid mass which was crystallized from methanol-dichloromethane mixture (20 mL, 1:3 v/v). The yellow colored crystals were formed. Yield: 60%; m.p. 155-57 °C. Anal. Calc. for AgClC₂₃H₂₀NSP: C, 53.43; H, 3.87; N, 2.71. Found: C, 53.78; H, 3.70; N, 3.01%. Main IR peaks (KBr, cm^{-1}), v(N-H), 3178(m); v(C-H), 3035–2920(m); v(C=N) + v(C-C); 1608(m)-1571(s); $\delta(N-H) + \delta(C-H),$ $1500(s)-1363(s); v(C=S), 1130(s); v(P-C_{Ph}) 1093(m).$ ¹H NMR (δ , CDCl₃), $\delta = 7.81(d, H^6)$, 7.26–7.63m (Ph₃P + $C^{3,4}H$; 6.89(t, H⁵). ³¹P NMR (CDCl₃), $\delta = -98.4$ ppm, $\Delta\delta(\delta_{\text{complex}} - \delta_{\text{PPh}_3}) = 14.8 \text{ ppm. } \delta P, -78.63, \Delta\delta = 34.80 \text{ ppm.}$

2.3. Synthesis of $[Ag_2(\mu-S-pySH)_2(PPh_3)_2Br_2]$ (2)

To AgBr (0.025 g, 0.13 mmol) suspended in 20 mL of acetonitrile was added solid PPh₃ (0.070 g, 0.26 mmol)

and stirring was carried out for 24 h. Acetonitrile was removed from the white colored precipitates and chloroform (15 mL) was added. To the precipitates suspended in CHCl₃ solid pyridine-2-thione (0.019 g, 0.17 mmol) was added and the contents were stirred for 1 h until a clear solution was obtained. Slow evaporation of solution at room temperature formed a solid mass which was treated with diethyl ether to remove any unreacted PPh₃ and then crystallized from methanol-dichloromethane mixture (25:75:v/v). The yellow colored crystals were formed. Yield: 65%; m.p. 190-195 °C. Anal. Calc. for AgBrC23-H₂₀NSP: C, 49.22; H, 3.55; N, 2.49. Found: C, 49.50; H, 3.91; N, 2.49%. Main IR peaks (KBr, cm⁻¹); v(N-H), 3170(m); v(C-H) 3058(m)-3035(m); v(C=N) + v(C=C); 1608(m) - 1569(s): $\delta(N-H) + \delta(C-H)$ 1498(s) - 1361(s);v(C=N), 1608(w); v(C=S), 1128(s); $v(P-C_{Ph})$, 1093(m). ¹H NMR data (δ , CDCl₃): 7.78 (d, H⁶); 7.67 (m, H⁴); 7.57(d, H^3); 6.91(t, H^5); 7.32–7.53 (PPh₃). ³¹P NMR (CDCl₃), $\delta = -100.6$, $\Delta\delta(\delta_{\text{complex}} - \delta_{\text{PPh}_3}) = 12.6$ ppm. δP , $-78.29, \Delta \delta = 34.86$ ppm.

2.4. Crystallography

Single crystals of compounds 1 and 2 were mounted on CCD area detector diffractometer, equipped with a graphite monochromator and Mo K α radiator ($\lambda = 0.71073$ Å). The unit cell dimensions and intensity data were measured at 93(2) K. The structures were solved by the direct methods, and refined by the full matrix least square based on F^2 with anisotropic thermal parameters for the non-hydrogen atoms using Bruker SMART (data collection and cell refinement), Bruker SAINT (data reduction) SHELXS-97 (structure solution), and SHELXL-97 (structure refinement) and Bruker SHELXTL (molecular graphics) [13–16]. A multi-scan absorption correction (SADABS) was applied.

3. Results and discussion

3.1. Synthesis and IR spectroscopy

Reaction of silver(I) chloride with PPh₃ in acetonitirile and then with pyridine-2-thione chloroform (1:1:1 molar ratio) yielded compound of stoichiometry, {AgCl (pySH)(PPh₃)} and similar reaction with silver(I) bromide yielded, {AgBr(pySH)(PPh₃)}. The X-ray crystallography has shown both compounds to be dimers, $[Ag_2Cl_2(\mu-S-pySH)_2(PPh_3)_2]$ 1 and $[Ag_2Br_2(\mu-S-pySH)_2(PPh_3)_2]$ 2 (Scheme 1).

Complexes showed v(N-H) peaks at 3178 cm⁻¹ in 1 and at 3170 cm⁻¹ in 2, and it revealed that there is no deprotonation of NH proton of pySH ligand. The diagnostic

$$2AgX + 2PPh_3 + 2$$

 H
 H
 H
 H
 CH_3CN
 $CHCl_3$
 $(Ag_2X_2(\mu-S-pySH)_2(PPh_3)_2]$
 $X = Cl 1, Br 2$
Scheme 1.

v(C=S) peak in free pySH ligand occurs at 1138 cm⁻¹ [12], which is shifted to lower energy region in complexes, at 1130 cm⁻¹ **1** and 1128 cm⁻¹ **2**. These trends in v(C=S)and v(N-H) peaks suggest coordination by thione sulfur donor atom. The v(C-H) peaks fall in the regions 3055– 2920 cm⁻¹; while other peaks due to $v(C-C) + v(C=N) + \delta(N-H) + \delta(C-H)$ fall in the region, 1332–1608 cm⁻¹. A characteristic peak due to $v(P-C_{Ph})$ at 1093 cm⁻¹ indicated the presence of PPh₃ in the complexes.

3.2. Crystal structures of the complexes

Compounds $[Ag_2(\mu$ -S-pySH)₂Cl₂(PPh₃)₂] **1** and $[Ag_2(\mu$ -S-pySH)₂Br₂(PPh₃)₂] **2** crystallized as triclinic crystals in space group $P\bar{1}$. The atomic numbering schemes of complexes **1** and **2** are shown in Figs. 1 and 2, respectively. Crystal data are given in Table 1 and bond lengths and angles are given in Table 2.

In complex 1, Ag is bonded to one Cl atom, one P atom of Ph₃P and one S atom of pySH forming three coordinate unit, $\{AgCl(Ph_3P)(pvSH)\}$, and two such units dimerize via the bonded S atoms to yield sulfur-bridged dimer $[Ag_2Cl_2(\mu-S-pySH)_2(PPh_3)_2]$ (1) (Fig. 1). The formation of $[Ag_2Br_2(\mu-S-pySH)_2(PPh_3)_2]$ (2) (Fig. 2) is similar with Br as the anion. The central Ag_2S_2 core forms a parallelogram with two unequal Ag-S bond distances (2.5832(8), 2.7208(11) Å) in 1 and (2.6306(4), 2.6950(7) Å) in 2, respectively. These bridging Ag–S bond distances are in the range of terminal Ag-S bond distance, 2.625 Å in [AgCl-(pySH)(PPh₃)₂] [9a]. The S-Ag-S and Ag-S-Ag bond angles within the {Ag(μ -S)₂Ag} core of 1 are 87.190(15)° and, 92.810(15)° 1, respectively. The similar angles are 87.814(18)° and 92.187(18)° in 2. The angles around Ag vary in the range 87.19(2)-121.71(2)° in 1, and 87.81(2)- $121.53(2)^{\circ}$ in 2, which indicate highly distorted tetrahedral geometries. Finally the Ag–S–C bond angles are $105.10(6)^{\circ}$,



Fig. 1. Structure of complex 1 showing the atomic numbering scheme.



Fig. 2. Structure of complex 2 showing the atomic numbering scheme.

Table 1		
Crystal data	and refinement details for complexes 1 and 2	

	1	2
Empirical formula	$C_{46}H_{40}Ag_2Cl_2N_2P_2S_2$	$C_{46}H_{40}Ag_2Br_2N_2P_2S_2$
M	1033.50	1122.42
$T(\mathbf{K})$	93(2)	93(2)
Crystal system	triclinic	triclinic
Space group	$P\overline{1}$	$P\overline{1}$
Unit cell dimensions		
a (Å)	9.520(3)	9.526(1)
$b(\mathbf{A})$	9.507(3)	9.583(1)
$c(\mathbf{A})$	14.014(5)	14.121(2)
α (°)	71.563(5)	77.512(2)
β (°)	77.357(6)	72.176(2)
γ (°)	62.924(5)	63.011(2)°
$V(\dot{A}^3)$	1067.1(6)	1088.9(3)
Ζ	1	1
$D_{\text{calc}} (\text{mg m}^{-3})$	1.608	1.712
$\mu (\mathrm{mm}^{-1})$	1.251	2.938
Reflections collected	8376	8192
Unique reflections [R(int)]	5003 [0.0268]	5049 [0.0161]
Reflections with $[I > 2\sigma(I)]$	4807	4438
Final <i>R</i> indices $[I > 2\sigma(I)]$	$R_1 = 0.0254, wR_2 = 0.0665$	$R_1 = 0.0235, wR_2 = 0.0562$
R indices (all data)	$R_1 = 0.0267, wR_2 = 0.0673$	$R_1 = 0.0306, wR_2 = 0.0600$

Table 2

Selected bond length (A) and angles(°) for	1 and 2
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1			
Ag–P	2.435(1)	Ag–S	2.721(1)
Ag–Cl	2.530(1)	Ag–S	$2.583(1)^{\#1}$
S-C(1)	1.722(2)	$Ag \cdots Ag$	3.8425(8)
P-Ag-Cl	121.71(2)	P–Ag–S	101.35(2)
Cl–Ag–S	103.91(2)	P–Ag–S ^{#1}	120.70(2)
Cl-Ag-S ^{#1}	112.24(3)	Ag–S–Ag	92.81(2)
Ag-S-C(1)	109.93(6)	S–Ag–S	87.19(2)
$Ag^{\#1}-S-C(1)$	105.10(6)		
2			
Ag–P	2.441(1)	Ag–Br	2.631(1)
Ag–S	2.608(1)	Ag–S ^{#1}	2.695(1)
S-C(1)	1.717(2)	Ag···Ag	3.8211(4)
P–Ag–Br	121.53(2)	P–Ag–S	121.16(2)
S–Ag–Br	109.47(2)	P–Ag–S ^{#1}	102.44(2)
$Br_\Delta \sigma_S^{\#1}$	10(00(1)		02.10(2)
DI-Ag-5	106.98(1)	Ag–5–Ag	92.19(2)
Ag-S-C(1)	108.98(1) 103.79(8)	Ag-S-Ag S-Ag-S ^{#1}	92.19(2) 87.81(2)

 $109.93(6)^{\circ}$ in **1** and $103.79(8)^{\circ}$, $111.62(1)^{\circ}$ in **2** and are similar to that reported for [Ag(pySH)(PPh₃)₂Cl] [9a].

The Ag–Cl bond is strong as the bond distance in complex 1, 2.530(1) Å is less than the sum of the ionic radii of Ag⁺ and Cl⁻, 2.7501 Å [17]. The Ag–Br bond distance in complex 2, 2.631(1) Å, is shorter than the Ag–Br bridging bond distance [2.7350(6), 2.8241(5) Å] observed in [Ag₂Br₂(μ -S-pymSH)₂(PPh₃)₂] [10a]. The S–C bond distance 1.722(2) Å in 1 is slightly larger than 1.717(2) Å in complex 2. This is due to high electronegativity of Cl⁻ versus Br⁻ which weakens C–S bond in the former. The Ag–P bond distances are, 2.4346(10) Å in 1 and 2.4414(6) Å in 2, which are nearly same. The Ag…Ag contacts of compounds 1 and 2 of 3.8425(8) and 3.8211(4) Å, respectively, are higher than twice the sum of van der Waals radius of Ag atom, 3.40 Å.

There are inter dimer π - π stacking interactions between pyridyl rings. The inter ring distances are 3.498 and 3.510 Å in complexes **1** and **2**, respectively. Packing diagrams are shown in Figs. 3 and 4.

3.3. NMR spectroscopy

Proton NMR spectra of sparingly soluble complexes, $[AgCl(pySH)(PPh_3)]_2$ 1 and $[AgBr(pySH)(PPh_3)]_2$ 2, in CDCl₃ has been recorded. Complexes 1 and 2 show peaks due to H⁶ proton at low field, δ 7.81 and 7.78 ppm, respectively, vis-à-vis the free ligand (pySH) at δ 7.56 ppm [12]. Likewise, H^5 proton signals at δ 6.89 (1) and 6.91 (2) ppm are downfield (free pySH, δ 6.73 ppm). The H³ and H^4 proton signals for 1 merged with the PPh₃ signals in the region, δ 7.26–7.63 ppm; while in complex **2**, these signals were clearly visible (see Section 2). The ³¹P NMR spectra showed two peaks for each complex with coordination shifts, 14.8, 34.80 ppm (1) and 12.6, 34.86 (2) ppm. These data reveal that dimers (δ 34.80, 34.86 ppm 1 and 2) probably set an equilibrium with monomers (14.8, 12.6 ppm, 1a, 2a, respectively) in solution. The monomers are suggested as $AgX(pySH)(PPh_3)_2$ (X = Cl, 1a; Br, 2a) formed by rearrangement of dimers in solution, a phenomenon observed in copper(I) halide-thiosemicarbazone chemistry [18].

3.4. Electronic absorption spectroscopy

The electronic absorption spectrum of complex 1 in dichloromethane showed three peaks at λ_{max} values of 238, 270 and 359 nm. Similarly complex 2 has shown two peaks at λ_{max} values of 239, 271 and 359 nm. The peaks



Fig. 3. Packing diagram of complex 1.



Fig. 4. Packing diagram of complex 2.

at 359 nm are attributed to -C=S groups due to $n \to \pi^*$ transitions while other beaks are due to $\pi \to \pi^*$ transitions based on pyridyl and phenyl rings. Neither of the two compounds exhibited any fluorescence properties.

3.5. Conclusion

Pyridine-2-thione has formed hitherto unknown dimers **1** and **2**. It may be noted that AgCl has formed both previously known monomer, $[Ag(pySH)(PPh_3)_2Cl]$ [9a], and the newly formed dimer $[Ag_2Cl_2(\mu$ -S-pySH)_2(PPh_3)_2]

1; while for copper(I) chloride only monomer, $[CuCl(pySH)-(PPh_3)_2]$ is known to date as all efforts to get a dimer $[Cu_2Cl_2 (\mu-S-pySH)_2(PPh_3)_2]$ ended in the formation of only monomer $[CuCl(pySH)(PPh_3)_2]$ due to strong intramolecular – NH···Cl hydrogen bonding in the latter [12].

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Appendix A. Supplementary data

CCDC 657505 and 657504 contain the supplementary crystallographic data for 1 and 2. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/ conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033, or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2008.01.008.

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