Crystal structure of tris(triphenylphosphine)bis(μ-phenyl-selenolato)di-silver(I), $Ag_2[P(C_6H_5)_3]_3(C_6H_5Se)_2$

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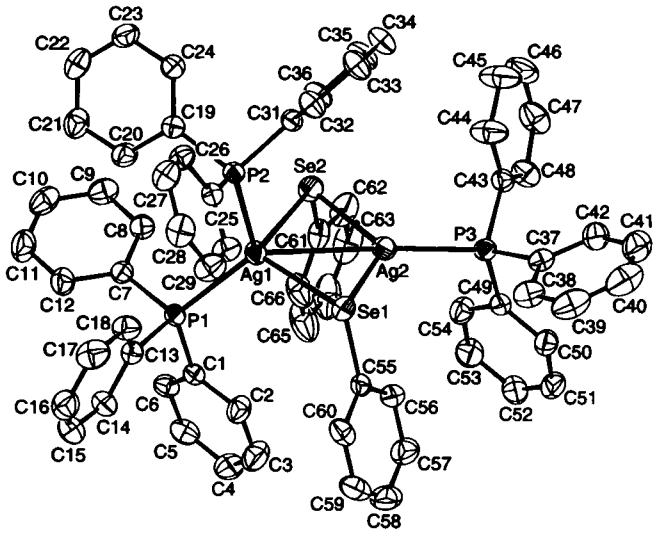
Received October 19, 2004, accepted and available on-line December 31, 2004; CCDC no. 1267/1410



Abstract

$C_{66}H_{55}Ag_2P_3Se_2$, triclinic, $P\bar{1}$ (no. 2), $a = 11.0749(6)$ Å, $b = 13.4572(7)$ Å, $c = 20.501(1)$ Å, $\alpha = 80.666(1)°$, $\beta = 86.561(1)°$, $\gamma = 72.516(1)°$, $V = 2875.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.038$, $wR_{ref}(F^2) = 0.090$, $T = 294$ K.

Source of material

The reactions were carried out under a nitrogen atmosphere using standard Schlenk techniques. All solvents were dried and freshly distilled. Benzeneselenol was prepared by the modified literature procedure [1]. At room temperature, a solution of silver nitrate (0.085 g, 0.5 mmol) in acetonitrile (5 ml) was added to a mixture of benzeneselenol (0.15 ml, 1.5 mmol) and triethylamine (0.24 ml, 1.5 mmol) in acetonitrile (5.0 ml). Then, triphenylphosphine (0.262 g, 1.0 mmol) in methanol (15 ml) was added with brief stirring to dissolve any precipitate. Crystals were obtained by slow evaporation of an acetonitrile and methanol solution of the compound (m.p. 384-387 K).

Discussion

Increasing interest in the investigation of silver(I) complexes with chalcogenide ligands has emerged [2]. The study of silver chalcogenide complexes attracts wide interest not only owing to their diversity of bonding modes and structural motifs, but also for their potential use in biological systems and as precursors for materials [3].

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Here we reported the neutral dinuclear silver(I) with Se, the P-contained ligand complex structure of $[Ag_2(\mu\text{-PhSe})_2(PPh_3)_3]$, in which two Se atoms of the selenophenol ligands bridge two Ag atoms to form a quadrilateral. The two Ag atoms have different coordination. Ag2 is planar trigonally coordinated by two bridge Se atoms and one P atom of PPh_3, while Ag1 is coordinated by two bridge Se atoms and two P atoms of PPh_3 to form a distorted tetrahedron. The Se—Ag bonds of Ag1 atom (2.7889 Å and 2.8011 Å) are significantly longer than those of Ag2 atom (2.5924 Å and 2.6171 Å), and the P—Ag bonds for Ag1 atom (2.4879 Å and 2.5418 Å) are longer than that for Ag2 (2.4553 Å). The reason is that the coordination number of Ag1 is greater than that of Ag2. The Ag—Ag distance is 3.0250(4) Å, which is shorter than the sum of the van-der-Waals radii of two Ag atoms (3.44 Å), and longer than the Ag—Ag distance (2.866 Å) in metallic Ag, and may suggest they are related by so-called argentophilicity [4]. Similar structures are known for copper(I) complexes [5,6].

Table 1. Data collection and handling.

Crystal:	yellow, columnar, size 0.1 × 0.15 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	20.70 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.7°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	33841, 14212
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 10263
$N(param)_{refined}$:	658
Programs:	SHELXS-97 [7], SHELXL-97 [8]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	2*i*	0.3030	0.2324	0.1102	0.077
H(3A)	2*i*	0.3560	0.1834	0.0074	0.097
H(4A)	2*i*	0.4193	0.2947	−0.0758	0.085
H(5A)	2*i*	0.4181	0.4587	−0.0579	0.074
H(6A)	2*i*	0.3567	0.5122	0.0438	0.063
H(8A)	2*i*	0.4861	0.4687	0.2042	0.067
H(9A)	2*i*	0.5289	0.6247	0.2112	0.081
H(10A)	2*i*	0.3779	0.7836	0.1828	0.088
H(11A)	2*i*	0.1839	0.7886	0.1466	0.095
H(12A)	2*i*	0.1397	0.6339	0.1388	0.079
H(14A)	2*i*	0.0849	0.4824	0.0675	0.072
H(15A)	2*i*	−0.1316	0.5272	0.0658	0.090
H(16A)	2*i*	−0.2447	0.5172	0.1624	0.094

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(17A)	2*i*	–0.1412	0.4598	0.2616	0.096
H(18A)	2*i*	0.0777	0.4132	0.2641	0.077
H(20A)	2*i*	0.2200	0.5121	0.3096	0.071
H(21A)	2*i*	0.2068	0.6823	0.3214	0.083
H(22A)	2*i*	0.2938	0.7150	0.4118	0.080
H(23A)	2*i*	0.3859	0.5784	0.4929	0.079
H(24A)	2*i*	0.3930	0.4082	0.4838	0.067
H(26A)	2*i*	0.1283	0.3886	0.4826	0.067
H(27A)	2*i*	–0.0713	0.3787	0.5132	0.081
H(28A)	2*i*	–0.1678	0.2902	0.4563	0.088
H(29A)	2*i*	–0.0653	0.2105	0.3695	0.090
H(30A)	2*i*	0.1381	0.2155	0.3401	0.072
H(32A)	2*i*	0.2857	0.1751	0.5070	0.070
H(33A)	2*i*	0.4238	0.0666	0.5865	0.083
H(34A)	2*i*	0.6365	0.0467	0.5759	0.082
H(35A)	2*i*	0.7122	0.1377	0.4854	0.086
H(36A)	2*i*	0.5753	0.2452	0.4056	0.073
H(38A)	2*i*	0.5654	–0.1756	0.2919	0.087
H(39A)	2*i*	0.5350	–0.3359	0.3370	0.112
H(40A)	2*i*	0.7007	–0.4706	0.3850	0.121
H(41A)	2*i*	0.8978	–0.4494	0.3857	0.110

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(42A)	2*i*	0.9309	–0.2920	0.3396	0.083
H(44A)	2*i*	0.7978	–0.0441	0.3957	0.107
H(45A)	2*i*	0.9558	–0.0299	0.4578	0.137
H(46A)	2*i*	1.1515	–0.0423	0.4125	0.111
H(47A)	2*i*	1.1963	–0.0830	0.3075	0.100
H(48A)	2*i*	1.0417	–0.1103	0.2471	0.078
H(50A)	2*i*	0.8398	–0.2644	0.1940	0.075
H(51A)	2*i*	0.8980	–0.2772	0.0848	0.093
H(52A)	2*i*	0.9234	–0.1338	0.0147	0.091
H(53A)	2*i*	0.8910	0.0233	0.0524	0.087
H(54A)	2*i*	0.8270	0.0388	0.1601	0.074
H(56A)	2*i*	0.5384	–0.0042	0.1670	0.069
H(57A)	2*i*	0.5297	–0.0599	0.0677	0.090
H(58A)	2*i*	0.3387	–0.0590	0.0283	0.107
H(59A)	2*i*	0.1582	–0.0031	0.0891	0.098
H(60A)	2*i*	0.1644	0.0543	0.1884	0.075
H(62A)	2*i*	0.8997	0.2255	0.2177	0.096
H(63A)	2*i*	0.9932	0.2352	0.1131	0.126
H(64A)	2*i*	0.8743	0.2633	0.0215	0.135
H(65A)	2*i*	0.6644	0.2779	0.0308	0.138
H(66A)	2*i*	0.5681	0.2679	0.1339	0.104

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	2*i*	0.38394(2)	0.27636(2)	0.26472(1)	0.0528(2)	0.0454(1)	0.0393(1)	–0.0046(1)	0.0013(1)	–0.00380(9)
Ag(2)	2*i*	0.59129(2)	0.06837(2)	0.27668(1)	0.0483(1)	0.0573(2)	0.0487(1)	–0.0115(1)	–0.0009(1)	–0.0106(1)
Se(1)	2*i*	0.35510(3)	0.07576(2)	0.27326(2)	0.0466(2)	0.0472(2)	0.0537(2)	–0.0161(1)	0.0078(1)	–0.0113(1)
Se(2)	2*i*	0.64439(3)	0.24599(3)	0.27324(2)	0.0499(2)	0.0582(2)	0.0684(2)	–0.0172(2)	0.0019(2)	–0.0154(2)
P(1)	2*i*	0.27709(7)	0.40883(6)	0.17063(4)	0.0487(4)	0.0394(4)	0.0374(4)	–0.0110(3)	–0.0028(3)	–0.0021(3)
P(2)	2*i*	0.31332(7)	0.30895(6)	0.38165(4)	0.0395(4)	0.0456(4)	0.0344(4)	–0.0102(3)	0.0014(3)	–0.0064(3)
P(3)	2*i*	0.77064(7)	–0.09092(6)	0.27250(4)	0.0399(4)	0.0480(4)	0.0479(4)	–0.0131(3)	–0.0010(3)	–0.0111(3)
C(1)	2*i*	0.3251(3)	0.3771(2)	0.0884(1)	0.047(2)	0.043(2)	0.038(2)	–0.010(1)	–0.006(1)	–0.003(1)
C(2)	2*i*	0.3254(4)	0.2789(2)	0.0764(2)	0.093(3)	0.045(2)	0.051(2)	–0.018(2)	0.008(2)	–0.005(2)
C(3)	2*i*	0.3583(4)	0.2491(3)	0.0150(2)	0.113(3)	0.060(2)	0.068(3)	–0.018(2)	0.011(2)	–0.022(2)
C(4)	2*i*	0.3946(4)	0.3158(3)	–0.0349(2)	0.078(3)	0.085(3)	0.048(2)	–0.015(2)	0.009(2)	–0.023(2)
C(5)	2*i*	0.3942(3)	0.4132(3)	–0.0240(2)	0.058(2)	0.082(3)	0.044(2)	–0.027(2)	0.001(2)	0.001(2)
C(6)	2*i*	0.3584(3)	0.4451(3)	0.0371(2)	0.056(2)	0.059(2)	0.045(2)	–0.023(2)	–0.004(2)	–0.001(2)
C(7)	2*i*	0.3073(3)	0.5350(2)	0.1713(1)	0.057(2)	0.044(2)	0.037(2)	–0.015(1)	0.001(1)	–0.004(1)
C(8)	2*i*	0.4246(3)	0.5329(3)	0.1926(2)	0.058(2)	0.056(2)	0.053(2)	–0.015(2)	–0.000(2)	–0.009(2)
C(9)	2*i*	0.4502(4)	0.6264(3)	0.1968(2)	0.075(3)	0.073(2)	0.066(2)	–0.039(2)	0.006(2)	–0.014(2)
C(10)	2*i*	0.3602(4)	0.7212(3)	0.1797(2)	0.102(3)	0.056(2)	0.074(3)	–0.043(2)	0.012(2)	–0.015(2)
C(11)	2*i*	0.2448(4)	0.7242(3)	0.1582(2)	0.089(3)	0.043(2)	0.101(3)	–0.017(2)	–0.005(3)	–0.003(2)
C(12)	2*i*	0.2184(4)	0.6313(3)	0.1538(2)	0.070(2)	0.046(2)	0.079(2)	–0.015(2)	–0.013(2)	–0.003(2)
C(13)	2*i*	0.1054(3)	0.4435(2)	0.1659(2)	0.049(2)	0.043(2)	0.049(2)	–0.012(1)	–0.001(1)	–0.007(1)
C(14)	2*i*	0.0400(3)	0.4776(3)	0.1071(2)	0.058(2)	0.063(2)	0.055(2)	–0.013(2)	–0.008(2)	–0.002(2)
C(15)	2*i*	–0.0897(4)	0.5046(3)	0.1059(2)	0.055(2)	0.077(3)	0.084(3)	–0.007(2)	–0.016(2)	–0.010(2)
C(16)	2*i*	–0.1567(4)	0.4984(3)	0.1632(2)	0.051(2)	0.075(3)	0.107(3)	–0.008(2)	–0.001(2)	–0.029(2)
C(17)	2*i*	–0.0950(4)	0.4643(4)	0.2223(2)	0.066(3)	0.105(3)	0.076(3)	–0.028(2)	0.022(2)	–0.036(2)
C(18)	2*i*	0.0362(3)	0.4366(3)	0.2238(2)	0.061(2)	0.078(2)	0.057(2)	–0.023(2)	0.001(2)	–0.017(2)
C(19)	2*i*	0.3088(3)	0.4409(2)	0.3953(1)	0.040(2)	0.052(2)	0.041(2)	–0.014(1)	0.007(1)	–0.009(1)
C(20)	2*i*	0.2531(3)	0.5248(3)	0.3470(2)	0.079(2)	0.056(2)	0.043(2)	–0.019(2)	–0.000(2)	–0.009(2)
C(21)	2*i*	0.2461(4)	0.6268(3)	0.3537(2)	0.090(3)	0.051(2)	0.060(2)	–0.015(2)	0.010(2)	–0.003(2)
C(22)	2*i*	0.2968(3)	0.6464(3)	0.4078(2)	0.064(2)	0.053(2)	0.089(3)	–0.025(2)	0.022(2)	–0.024(2)
C(23)	2*i*	0.3517(3)	0.5650(3)	0.4561(2)	0.054(2)	0.070(2)	0.082(3)	–0.017(2)	–0.006(2)	–0.035(2)
C(24)	2*i*	0.3569(3)	0.4628(3)	0.4503(2)	0.051(2)	0.056(2)	0.061(2)	–0.012(2)	–0.005(2)	–0.014(2)
C(25)	2*i*	0.1545(3)	0.3039(2)	0.4078(1)	0.042(2)	0.043(2)	0.042(2)	–0.010(1)	0.002(1)	–0.004(1)
C(26)	2*i*	0.0901(3)	0.3520(3)	0.4597(2)	0.052(2)	0.061(2)	0.057(2)	–0.017(2)	0.011(2)	–0.020(2)
C(27)	2*i*	–0.0294(3)	0.3465(3)	0.4779(2)	0.059(2)	0.073(2)	0.071(2)	–0.018(2)	0.026(2)	–0.023(2)
C(28)	2*i*	–0.0867(3)	0.2935(3)	0.4441(2)	0.051(2)	0.076(2)	0.097(3)	–0.027(2)	0.020(2)	–0.013(2)
C(29)	2*i*	–0.0256(4)	0.2457(3)	0.3926(2)	0.063(2)	0.087(3)	0.091(3)	–0.039(2)	0.010(2)	–0.029(2)
C(30)	2*i*	0.0958(3)	0.2495(3)	0.3746(2)	0.053(2)	0.073(2)	0.063(2)	–0.026(2)	0.009(2)	–0.023(2)
C(31)	2*i*	0.4150(3)	0.2222(2)	0.4478(1)	0.043(2)	0.047(2)	0.040(2)	–0.008(1)	–0.003(1)	–0.007(1)
C(32)	2*i*	0.3719(3)	0.1679(3)	0.5022(2)	0.049(2)	0.068(2)	0.051(2)	–0.015(2)	0.001(2)	0.005(2)
C(33)	2*i*	0.4547(4)	0.1026(3)	0.5500(2)	0.068(3)	0.076(2)	0.053(2)	–0.019(2)	–0.005(2)	0.015(2)
C(34)	2*i*	0.5810(4)	0.0908(3)	0.5439(2)	0.064(2)	0.076(2)	0.056(2)	–0.013(2)	–0.019(2)	0.010(2)
C(35)	2*i*	0.6260(3)	0.1449(3)	0.4897(2)	0.048(2)	0.095(3)	0.067(2)	–0.018(2)	–0.013(2)	0.005(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(36)	2*i*	0.5439(3)	0.2095(3)	0.4421(2)	0.052(2)	0.075(2)	0.050(2)	−0.018(2)	−0.003(2)	0.006(2)
C(37)	2*i*	0.7516(3)	−0.2170(2)	0.3107(2)	0.056(2)	0.055(2)	0.046(2)	−0.024(2)	0.005(2)	−0.013(1)
C(38)	2*i*	0.6331(4)	−0.2307(3)	0.3104(2)	0.072(3)	0.085(3)	0.071(2)	−0.039(2)	0.008(2)	−0.018(2)
C(39)	2*i*	0.6147(5)	−0.3265(4)	0.3379(2)	0.112(4)	0.112(4)	0.089(3)	−0.078(3)	0.028(3)	−0.033(3)
C(40)	2*i*	0.7136(7)	−0.4069(4)	0.3661(2)	0.174(6)	0.087(3)	0.065(3)	−0.079(4)	0.030(3)	−0.012(2)
C(41)	2*i*	0.8307(5)	−0.3942(3)	0.3666(2)	0.142(5)	0.061(3)	0.071(3)	−0.032(3)	−0.006(3)	0.003(2)
C(42)	2*i*	0.8505(4)	−0.3000(3)	0.3391(2)	0.085(3)	0.057(2)	0.066(2)	−0.024(2)	−0.006(2)	−0.001(2)
C(43)	2*i*	0.9038(3)	−0.0807(2)	0.3155(2)	0.044(2)	0.046(2)	0.053(2)	−0.015(1)	−0.006(1)	−0.004(1)
C(44)	2*i*	0.8791(4)	−0.0546(4)	0.3779(2)	0.062(3)	0.146(4)	0.072(3)	−0.037(3)	−0.002(2)	−0.043(3)
C(45)	2*i*	0.9728(5)	−0.0436(5)	0.4146(2)	0.095(4)	0.178(6)	0.087(3)	−0.050(4)	−0.022(3)	−0.051(3)
C(46)	2*i*	1.0892(5)	−0.0526(4)	0.3882(3)	0.082(3)	0.101(3)	0.105(4)	−0.041(3)	−0.045(3)	−0.002(3)
C(47)	2*i*	1.1159(4)	−0.0768(3)	0.3257(2)	0.054(2)	0.092(3)	0.105(4)	−0.034(2)	−0.017(2)	0.012(3)
C(48)	2*i*	1.0230(3)	−0.0922(3)	0.2892(2)	0.053(2)	0.076(2)	0.068(2)	−0.026(2)	−0.005(2)	0.000(2)
C(49)	2*i*	0.8275(3)	−0.1111(2)	0.1891(1)	0.043(2)	0.045(2)	0.046(2)	−0.007(1)	−0.005(1)	−0.009(1)
C(50)	2*i*	0.8491(3)	−0.2056(3)	0.1657(2)	0.080(3)	0.047(2)	0.056(2)	−0.011(2)	−0.004(2)	−0.008(2)
C(51)	2*i*	0.8844(4)	−0.2134(3)	0.1003(2)	0.105(3)	0.059(2)	0.059(2)	−0.002(2)	−0.003(2)	−0.022(2)
C(52)	2*i*	0.8995(4)	−0.1280(3)	0.0585(2)	0.087(3)	0.074(3)	0.048(2)	0.003(2)	0.004(2)	−0.009(2)
C(53)	2*i*	0.8796(4)	−0.0346(3)	0.0807(2)	0.090(3)	0.069(2)	0.052(2)	−0.019(2)	0.005(2)	−0.001(2)
C(54)	2*i*	0.8424(3)	−0.0259(3)	0.1455(2)	0.074(2)	0.049(2)	0.061(2)	−0.019(2)	0.004(2)	−0.008(2)
C(55)	2*i*	0.3531(3)	0.0311(2)	0.1893(2)	0.051(2)	0.035(1)	0.051(2)	−0.012(1)	−0.008(1)	−0.001(1)
C(56)	2*i*	0.4607(3)	−0.0034(3)	0.1518(2)	0.059(2)	0.058(2)	0.055(2)	−0.013(2)	−0.006(2)	−0.016(2)
C(57)	2*i*	0.4557(4)	−0.0369(3)	0.0921(2)	0.097(3)	0.070(2)	0.057(2)	−0.019(2)	0.001(2)	−0.018(2)
C(58)	2*i*	0.3423(6)	−0.0366(3)	0.0686(2)	0.143(5)	0.077(3)	0.056(2)	−0.045(3)	−0.025(3)	−0.004(2)
C(59)	2*i*	0.2353(5)	−0.0031(3)	0.1047(2)	0.103(4)	0.078(3)	0.072(3)	−0.048(3)	−0.040(3)	0.016(2)
C(60)	2*i*	0.2390(3)	0.0311(3)	0.1646(2)	0.056(2)	0.060(2)	0.068(2)	−0.022(2)	−0.015(2)	0.012(2)
C(61)	2*i*	0.7220(3)	0.2482(2)	0.1870(2)	0.053(2)	0.042(2)	0.075(2)	−0.009(2)	0.013(2)	−0.009(2)
C(62)	2*i*	0.8509(4)	0.2368(3)	0.1804(2)	0.061(2)	0.067(2)	0.120(4)	−0.027(2)	0.020(2)	−0.028(2)
C(63)	2*i*	0.9071(5)	0.2425(4)	0.1175(3)	0.082(3)	0.072(3)	0.160(5)	−0.030(3)	0.061(4)	−0.026(3)
C(64)	2*i*	0.8364(6)	0.2586(4)	0.0632(3)	0.117(5)	0.072(3)	0.112(4)	0.003(3)	0.051(4)	0.008(3)
C(65)	2*i*	0.7117(5)	0.2678(4)	0.0686(2)	0.104(4)	0.115(4)	0.076(3)	0.029(3)	0.018(3)	0.007(3)
C(66)	2*i*	0.6540(4)	0.2622(3)	0.1305(2)	0.063(3)	0.094(3)	0.076(3)	0.010(2)	0.008(2)	−0.002(2)

Acknowledgment. The authors gratefully acknowledge the financial support of the Natural Science Foundation of Guangdong Province (grant no. 990463).

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