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Silver–tin complex salts. A dimeric tin-complex anion with a double hydroxyl bridge. Crystal structure of $[Ag(AsPh_3)_4]_2[Sn_2Ph_4(NO_3)_4(OH)_2] \cdot 2CH_3CN$ *

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Abstract

A new silver–tin complex salt of formula $[Ag(AsPh_3)_4]_2[Sn_2Ph_4(NO_3)_4(OH)_2] \cdot 2CH_3CN$ has been synthesised by treating $Ag(AsPh_3)_2NO_3$ with stoichiometric amounts of $SnPh_3NO_3$ in acetone/acetonitrile. The crystal structure of the complex has been determined by X-ray diffraction. Of major interest is the dimeric structure of the anion with two tin atoms, related by a center of symmetry, linked through two bridging hydroxyl groups. In the dimeric unit each tin is in a distorted pentagonal bipyramidal environment, being also bound to three nitrate oxygens and two phenyl carbons. In the cation the coordination geometry around the silver atom is slightly distorted tetrahedral.

Introduction

In the course of our studies on the synthesis and structural characterization of organotin(IV) derivatives we have found that organotin chlorides and nitrates can form cationic [1], neutral [2,3] or anionic [4,5] complexes in which the coordination number of tin is increased from four to seven. The formation of complex salts containing tin complex anions seems to be strongly favoured when di- or triorganotin compounds are treated with silver salts in the presence of phosphine or arsine ligands. In the present paper the synthesis, the X-ray crystal structure, and the infrared spectrum of a new silver–tin complex salt of formula $[Ag(AsPh_3)_4]_2[Sn_2Ph_4(NO_3)_4(OH)_2] \cdot 2CH_3CN$ are reported and discussed.

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Experimental

Solvents were dried and distilled before use. $SnPh_3Cl$, $AsPh_3$, $AgNO_3$ were supplied commercially and were used without further purification. The preparation of $SnPh_3NO_3$ was reported previously [6,7].

$Ag(AsPh_3)_2NO_3$ – A solution of silver nitrate and triphenylarsine (1 : 2 molar ratio) in acetonitrile was stirred at room temperature for ca. 30 min. After some hours a white crystalline product was formed, and this was filtered and dried (m.p. 223 °C). The crude product was recrystallized from acetone/acetonitrile.

$[As(AsPh_3)_4]_2[Sn_2Ph_4(NO_3)_4(OH)_2].2CH_3CN$ – A solution of nitratotriphenyltin and nitratobis(triphenylarsine)silver (1 : 1 molar ratio) in an acetone/acetonitrile (1 : 1 v/v) mixture was stirred at room temperature exposed to the air for about 30 minutes. After several hours slow evaporation of the solvents in the air produced white crystals. Anal. Found: C, 57.9; H, 4.4; N, 2.2; Ag, 5.3; Sn, 6.1. $C_{86}H_{74}AgAs_4N_3O_7Sn$ calcd.: C, 57.8; H, 4.2; N, 2.4; Ag, 6.0; Sn, 6.6%.

Measurements. Elemental C, H, and N analyses were carried out with a Perkin–Elmer model 240 automatic analyser. Metal contents were determined by atomic absorption spectroscopy with a Perkin–Elmer model 303 instrument. Infrared spectra (4000–200 cm^{-1}) for KBr discs were recorded on a Perkin–Elmer model 283 B spectrophotometer.

Collection and reduction of X-ray data

X-ray crystallographic data were collected with a computer-controlled Philips PW 1100 four-circle diffractometer equipped with Mo-K_α radiation monochromated by graphite. A reciprocal-lattice search revealed no systematic extinctions or symmetry, indicating a triclinic lattice, the possible space groups being $P1$ and $P\bar{1}$. The choice of the latter was based on the statistical tests for the normalized structure factors, which showed the crystal to be centrosymmetric.

Crystal data: $C_{86}H_{74}AgAs_4N_3O_7Sn$, $M = 1787.79$, $a = 21.212(9)$, $b = 14.532(6)$, $c = 13.304(5)$ Å, $\alpha = 95.91(3)$, $\beta = 77.76(4)$, $\gamma = 75.47(3)$ °, $U = 3826(3)$ Å^3, $Z = 2$, $D_c = 1.552$ g cm^{-3}, $F(000) = 1792$, λ(Mo-K_α) = 0.71069 Å, μ(Mo-K_α) = 23.48 cm^{-1}.

Orientation and unit-cell parameters were determined from the angular positions of 30 intense carefully centered reflections distributed over a wide range of reciprocal space. Intensity data were recorded at room temperature up to $2\theta = 48$ °, using an ω–2θ scan technique with a scan rate of 0.075 ° s^{-1} and a scan width of 1.2 °. In all 12005 reflections (one hemisphere of data) were collected, 8565 of which were deemed observed according to the criterion $I > 3\sigma(I)$. There was no evidence of crystal decomposition or loss of alignment. The reflections were processed with the peak profile analysis procedure and corrected for Lorentz and polarization effects. A semiempirical absorption correction was also made on ψ scans of reflections with χ value near 90 ° (minimum and maximum transmission factors 1.000 and 1.186, respectively). A further correction was applied during the refinement by the empirical method of Walker and Stuart [8].

Solution and refinement of the structure

The positions of the tin, silver, and the four arsenic atoms were obtained by use of the automatic PATT routine in the SHELX-86 program package [9]. All the

Table 1

Fractional atomic coordinates ($\times 10^5$ for Sn, Ag, and As, $\times 10^4$ for O, N, and C)

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
Sn	55099(2)	38849(3)	50744(4)	C(36)	1956(2)	4654(3)	−1302(3)
Ag	20376(3)	22282(4)	13769(4)	C(37)	2340(2)	4847(3)	1219(3)
As(1)	33065(3)	11799(5)	6991(5)	C(38)	2821(2)	5146(3)	546(3)
As(2)	18851(3)	39442(5)	7104(5)	C(39)	3136(2)	5785(3)	942(3)
As(3)	18229(4)	25135(5)	34469(5)	C(40)	2971(2)	6124(3)	2012(3)
As(4)	11992(4)	12817(5)	8082(6)	C(41)	2490(2)	5824(3)	2685(3)
O(1)	4587(2)	4604(3)	4788(4)	C(42)	2175(2)	5186(3)	2289(3)
O(2)	6567(3)	3920(4)	5473(4)	C(43)	963(2)	4758(3)	1042(4)
O(3)	7523(3)	3869(6)	4470(6)	C(44)	459(2)	4290(3)	1168(4)
O(4)	6772(4)	5220(5)	5011(7)	C(45)	−215(2)	4826(3)	1450(4)
O(5)	5111(3)	2525(4)	4817(4)	C(46)	−385(2)	5831(3)	1605(4)
O(6)	5357(3)	1107(4)	5262(5)	C(47)	119(2)	6299(3)	1479(4)
O(7)	6036(3)	2004(4)	5294(4)	C(48)	793(2)	5763(3)	1197(4)
N(1)	6968(4)	4346(6)	4968(6)	C(49)	1367(3)	3845(3)	4054(4)
N(2)	5509(3)	1843(4)	5132(4)	C(50)	836(2)	4301(3)	3655(4)
N(3)	−1330(10)	2019(13)	5308(13)	C(63)	3658(2)	2367(3)	4238(4)
C(1)	5155(2)	3906(3)	6707(3)	C(51)	512(2)	5279(3)	3997(4)
C(2)	4654(2)	3471(3)	7073(3)	C(52)	720(2)	5802(3)	4739(4)
C(3)	4399(2)	3541(3)	8143(3)	C(53)	1252(2)	5346(3)	5139(4)
C(4)	4645(2)	4046(3)	8848(3)	C(54)	1575(2)	4368(3)	4796(4)
C(5)	5146(2)	4481(3)	8482(3)	C(55)	1236(2)	1860(4)	4208(4)
C(6)	5401(2)	4411(3)	7412(3)	C(56)	778(2)	2316(4)	5151(4)
C(7)	6117(3)	3443(4)	3512(3)	C(57)	379(2)	1815(4)	5708(4)
C(8)	6296(3)	4154(4)	2980(3)	C(58)	439(2)	859(4)	5322(4)
C(9)	6704(3)	3903(4)	1967(3)	C(59)	898(2)	404(4)	4380(4)
C(10)	6933(3)	2940(4)	1485(3)	C(60)	1296(2)	904(4)	3823(4)
C(11)	6755(3)	2229(4)	2017(3)	C(61)	2576(2)	2196(3)	4091(4)
C(12)	6347(3)	2480(4)	3031(3)	C(62)	3079(2)	2660(3)	3880(4)
C(13)	3569(2)	−56(3)	1170(4)	C(64)	3735(2)	1610(3)	4808(4)
C(14)	3848(2)	−927(3)	483(4)	C(65)	3231(2)	1145(3)	5019(4)
C(15)	4028(2)	−1788(3)	869(4)	C(66)	2652(2)	1439(3)	4660(4)
C(16)	3929(2)	−1776(3)	1941(4)	C(67)	1281(3)	8(3)	1203(4)
C(17)	3650(2)	−905(3)	2628(4)	C(68)	726(3)	−331(3)	1545(4)
C(18)	3470(2)	−44(3)	2243(4)	C(69)	815(3)	−1236(3)	1848(4)
C(19)	3912(2)	1842(3)	1169(4)	C(70)	1459(3)	−1802(3)	1810(4)
C(20)	4479(2)	1367(3)	1479(4)	C(71)	2014(3)	−1463(3)	1468(4)
C(21)	4871(2)	1893(3)	1860(4)	C(72)	1925(3)	−558(3)	1165(4)
C(22)	4695(2)	2894(3)	1930(4)	C(73)	1320(3)	973(4)	−693(3)
C(23)	4127(2)	3368(3)	1620(4)	C(74)	1484(3)	1651(4)	−1295(3)
C(24)	3736(2)	2842(3)	1239(4)	C(75)	1581(3)	1468(4)	−2375(3)
C(25)	3689(2)	850(4)	−800(3)	C(76)	1516(3)	608(4)	−2853(3)
C(26)	3339(2)	426(4)	−1398(3)	C(77)	1353(3)	−70(4)	−2251(3)
C(27)	3590(2)	173(4)	−2477(3)	C(78)	1255(3)	113(4)	−1171(3)
C(28)	4192(2)	345(4)	−2956(3)	C(79)	226(2)	1861(4)	1273(4)
C(29)	4542(2)	768(4)	−2358(3)	C(80)	−187(2)	1978(4)	580(4)
C(30)	4291(2)	1021(4)	−1279(3)	C(81)	881(2)	2367(4)	957(4)
C(31)	2169(2)	3881(3)	−797(3)	C(82)	−1161(2)	2641(4)	2028(4)
C(32)	2626(2)	3027(3)	−1366(3)	C(83)	−748(2)	2524(4)	2721(4)
C(33)	2870(2)	2945(3)	−2441(3)	C(84)	−55(2)	2135(4)	2344(4)
C(34)	2657(2)	3718(3)	−2946(3)	C(85)	−2429(10)	1725(13)	5201(14)
C(35)	2200(2)	4572(3)	−2376(3)	C(86)	−1814(9)	1865(12)	5238(13)

Table 2

Selected bond distances (Å) and angles (°)

Sn–O(1)	2.108(5)	N(2)–O(5)	1.298(8)	As(2)–C(31)	1.953(4)
Sn–O(1′) [a]	2.138(5)	N(2)–O(6)	1.211(10)	As(2)–C(37)	1.960(5)
Sn–O(2)	2.423(6)	N(2)–O(7)	1.255(10)	As(2)–C(43)	1.946(3)
Sn–O(5)	2.359(6)	Ag–As(1)	2.648(2)	As(3)–C(49)	1.933(4)
Sn–O(7)	2.752(6)	Ag–As(2)	2.698(1)	As(3)–C(55)	1.932(5)
Sn–C(1)	2.137(3)	Ag–As(3)	2.665(1)	As(3)–C(61)	1.947(5)
Sn–C(7)	2.137(4)	Ag–As(4)	2.685(1)	As(4)–C(67)	1.952(5)
N(1)–O(2)	1.291(11)	As(1)–C(13)	1.951(4)	As(4)–C(73)	1.948(4)
N(1)–O(3)	1.212(9)	As(1)–C(19)	1.950(5)	As(4)–C(79)	1.959(4)
N(1)–O(4)	1.225(12)	As(1)–C(25)	1.946(3)		
O(1)–Sn–O(1′) [a]	70.6(2)	O(2)–N(1)–O(4)	118.5(8)	Ag–As(1)–C(13)	118.0(2)
O(1′)–Sn–O(2)	79.7(2)	O(3)–N(1)–O(4)	122.5(9)	Ag–As(1)–C(19)	111.4(2)
O(2)–Sn–O(7)	77.8(2)	O(5)–N(2)–O(6)	119.7(7)	Ag–As(1)–C(25)	118.1(2)
O(7)–Sn–O(5)	49.5(2)	O(5)–N(2)–O(7)	116.5(6)	Ag–As(2)–C(31)	115.5(2)
O(5)–Sn–O(1)	82.6(2)	O(6)–N(2)–O(7)	123.8(7)	Ag–As(2)–C(37)	119.1(2)
C(1)–Sn–C(7)	160.2(2)	As(1)–Ag–As(2)	109.8(1)	Ag–As(2)–C(43)	114.8(2)
Sn–O(1)–Sn^i [a]	109.5(3)	As(1)–Ag–As(3)	104.4(1)	Ag–As(3)–C(49)	111.9(2)
Sn–O(2)–N(1)	122.7(5)	As(1)–Ag–As(4)	111.4(1)	Ag–As(3)–C(55)	117.1(2)
Sn–O(5)–N(2)	105.2(5)	As(2)–Ag–As(3)	109.1(1)	Ag–As(3)–C(61)	120.1(2)
Sn–O(7)–N(2)	87.6(5)	As(2)–Ag–As(4)	112.1(1)	Ag–As(4)–C(67)	116.2(2)
O(2)–N(1)–O(3)	119.0(9)	As(3)–Ag–As(4)	109.7(1)	Ag–As(4)–C(73)	114.6(2)
				Ag–As(4)–C(79)	120.1(2)

[a] $i = 1 - x, 1 - y, 1 - z$.

remaining non-hydrogen atoms, including a solvent molecule of acetonitrile, were found from successive structure factor and difference Fourier map calculations. The structure was refined by least-squares techniques to minimize the quantity $\Sigma w|\Delta F^2|$; unit weights were used in the first stages of refinement, while a weighting scheme was then introduced with $w = k[\sigma^2(F_o) + gF_o^2]^{-1}$. Anisotropic thermal parameters were used for all atoms except the carbon atoms in the cation and the atoms of the solvent molecule, which were refined isotropically. Due to the large number of refineable parameters, the anion and cation were allowed to vary alternately. All the fourteen phenyl rings were refined as rigid bodies of D_{6h} symmetry. No attempt was made to locate the hydrogen atoms. The final difference Fourier map was essentially featureless. The final residual indices for 376 variables refined were $R = 0.0469$ and $R_w = 0.0623$ with a GOF of 1.753.

Throughout the analysis atomic scattering factors for neutral atoms were used [10]. The calculations included both $\Delta f'$ and $\Delta f''$ corrections for the anomalous dispersion effects of the heavy atoms. Computations were performed on a GOULD-SEL 6040 computer using the programs of the SHELX-76 [11] and SHELX-86 [9] packages. The other programs used in the crystallographic work were as specified elsewhere [12].

Final atomic coordinates are listed in Table 1; selected bond distances and angles are given in Table 2. Lists of thermal parameters, observed and calculated structure factors, and a complete list of bond distances and angles are available from the authors.

Results and discussion

The crystal structure consists of discrete $[Sn_2Ph_4(NO_3)_4(OH)_2]^{2-}$ anions, $[Ag(AsPh_3)_4]^+$ cations, and solvating acetonitrile molecules. The geometries and atomic labelling schemes of the two complex ions are illustrated in Figs 1 and 2, respectively. The ionic nature of the complex salt is further confirmation of the ability shown by the tin atom to form anions.

The most interesting feature of the structure pertains to the dimeric formulation of the anion, with two tin atoms linked through two bridging hydroxyl groups. The presence of the hydroxyl groups can be understood in the light of the experimental conditions used (acetone solutions and exposure to the air), which could readily lead to some hydrolysis. The bridged Sn–O bonds are only slightly different, 2.108(5) and 2.138(5) Å. These values fall within the range of 2.047–2.169 Å found for hexacoordinate tin(IV) derivatives containing the Sn_2O_2 system [13–16]. The dimeric entities are centrosymmetric, with half a molecule comprising the crystallographic asymmetric unit and the other half being generated by an inversion center located in the middle of the dimer. To each tin atom are also coordinated three oxygen atoms from the two nitrate groups and two phenyl carbon atoms. As a consequence, in the dimeric unit the stereochemistry about each tin atom may be considered as distorted pentagonal bipyramidal, with the five oxygens occupying the equatorial positions and the two carbons at the apices. Some slight puckering of the girdle is observed, with a maximum displacement of 0.11 Å for the constituents

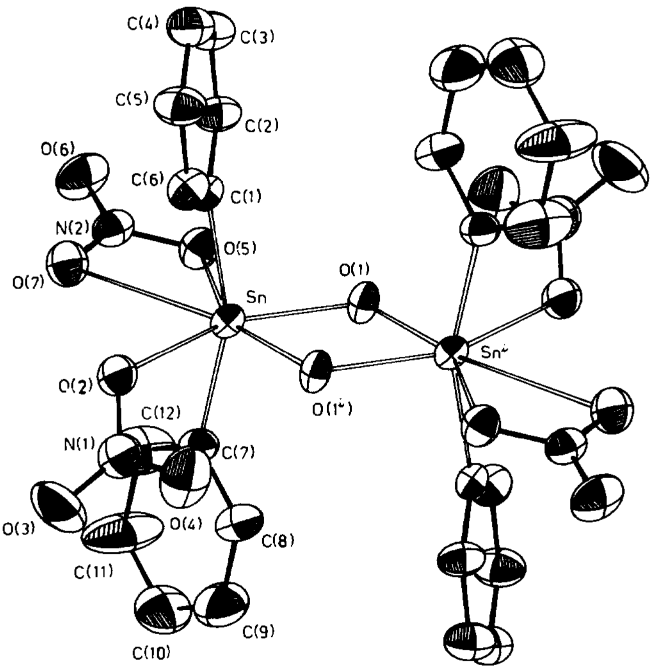

Fig. 1. ORTEP plot of the $[Sn_2Ph_4(NO_3)_4(OH)_2]^{2-}$ anion with thermal ellipsoids at the 40% probability level.

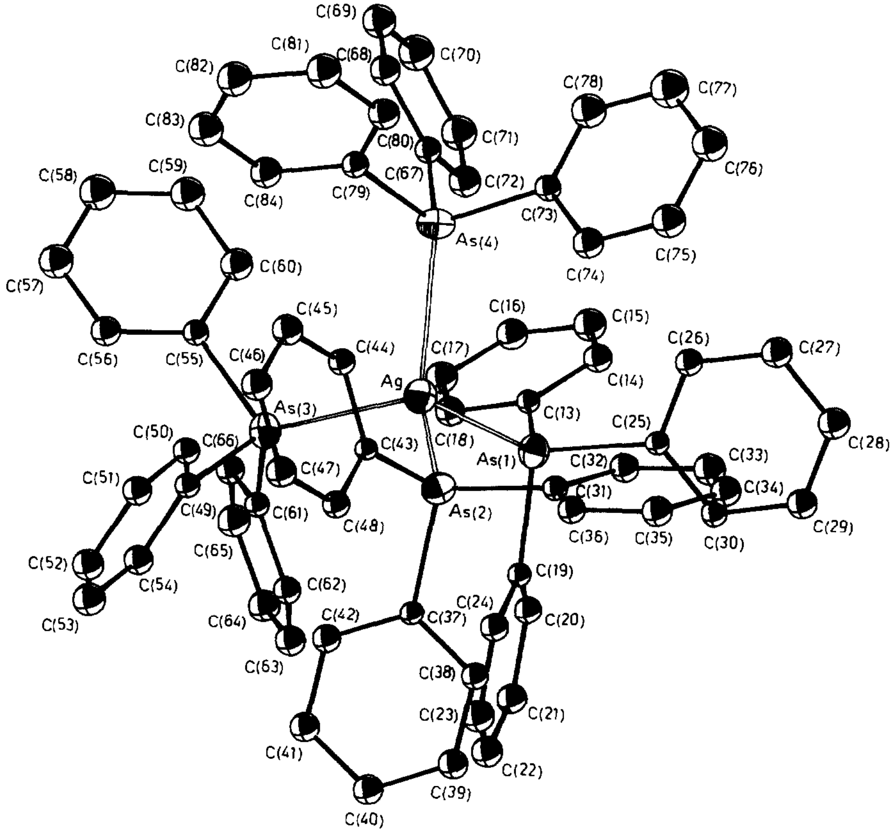

Fig. 2. ORTEP plot of the $[Ag(AsPh_3)_4]^+$ cation with the thermal ellipsoids at the 50% probability level.

atoms from the weighted mean plane. Deviation from regular pentagonal bipyramidal geometry mainly arises from the short bite of the bidentate nitrate group, which results in an O–Sn–O angle of 49.5(2)° rather than the 72° expected for an ideal geometry. The remaining four angles subtended at tin in the girdle range from 70.6(2) to 82.6(2)°. The irregularity of the pentagon can also be seen from the values of the five O ··· O contacts, namely 2.170(8), 3.261(9), 2.931(7), 2.452(8), and 2.954(7) Å.

This structure again illustrates the marked tendency of the diorgano groups to favour a pentagonal bipyramidal arrangement, as well as the versatility of the nitrate ion in coordination to metal. Concerning the latter point, in this compound there are two different kinds of nitrates, one of which binds the tin atom in a unidentate manner, Sn–O(2) 2.423(6) Å, while the other is chelated to metal with a short (Sn–O(5) 2.359(6) Å) and a long (Sn–O(7) 2.752(6) Å) bond, as already observed in other seven-coordinate nitrate diorganotin derivatives [5]. This kind of chelation can be classified as bidentate asymmetrical, in conformity with the statement of Addison et al. [17] that a metal–oxygen bond length difference of 0.2–0.7 Å for a bidentate nitrate constitutes unsymmetrical chelation. The values of the Sn–O(2)–N(1) 122.7(5)°; Sn–O(5)–N(2) 105.2(5)°, and Sn–O(7)–N(2) 87.6(5)° angles are in agreement with the different ligand behaviour of the two nitrates, as the angle at the coordinated oxygen tends to 110°, and is increased when the ligand is unidentate and decreased when is bidentate.

Within the four-membered Sn_2O_2 ring system, which is constrained to be planar by symmetry requirements, the angles are acute at tin, 70.6(2)°, and obtuse at oxygen, 109.5(3)°. Consistent with this, the two Sn atoms are separated by 3.463(2) Å across the parallelogram, while the O · · · O separation is only 2.452(8) Å.

The coordination geometry of the silver atom in the cation is that of a slightly distorted tetrahedron, with all four of the Ag–As distances of very similar length, 2.648(2)–2.698(1) Å and all the As–Ag–As angles lying between 104.4(1) and 112.1(1)°. The arsenic atoms show the expected distortion from tetrahedral symmetry, with the Ag–As–C angles (range: 111.4(2)–120.1(2)°) all larger than 109.5° and the C–As–C angles (range: 100.3(2)–103.7(2)°) all smaller. The As–C distances and the As–C–C angles have normal values, and average 1.948 Å and 120.0°, respectively. The geometrical parameters of the cation agree fairly well with the results for the only two other related compounds containing the same cation [18].

The molecular packing indicates only normal van der Waals forces between the two ions. The closest anion–cation non-hydrogen contact is 3.20(1) Å between O(3) of the asymmetric unit and C(53) at $1-x$, $1-y$, $1-z$ position. The solvent molecule is subject to relatively large thermal motion due to the absence of any significant close interaction with other species, the shortest contact being C(85) · · · O(7) ($x-1$, y, z) 3.15(2) Å.

The infrared spectrum of the dimer complex is mainly characterized by the vibrational absorptions of the phenyl rings and the nitrate groups. It matches fairly well that of the parent $Ag(AsPh_3)_2NO_3$, except for the two absorptions at 3570 and 565 cm^{-1}, which can be attributed to the stretching vibrations of the OH group [19] and the Sn–O bond [20–23], respectively (Table 3). Partial hydrolysis of the product occurred during the recording of the spectrum in a KBr disc can account for the

Table 3

Comparison of the main vibrational bands (cm^{-1}) of $Ag(AsPh_3)_2NO_3$ (I) and $[Ag(AsPh_3)_4]_2[Sn_2Ph_4(NO_3)_4(OH)_2]\cdot 2CH_3CN$ (II)

(I)	(II)	Assignment
–	3570 m	ν (OH)
1575 m	1575 m	ring
1480 ms	1480 ms	ring
1430 s	1430 s	ring
1390 vs	1380 vs	ν (NO_3)
1300 vs	1360 sh	
1075 ms	1072 ms	ring and
1020 ms	1020 ms	ν (NO_3)
995 ms	995 ms	in plane
840 mw	–	ν (NO_3)
820 mw	820 mw	out of plane
745 s	745 s	
735 vs	735 vs	δ (CH)
690 vs	690 vs	
–	565 m	ν (SnO)

intense band at 1380 cm^{-1} characteristic of an ionic nature [24–26], which has not been found from the X-ray structure.

Extensive use of the Cambridge Crystallographic Database Files was made during the bibliographic search.

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