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First synthesis and structural report on selenophen-2-yl containing pnictogens: Biological activities of tris(selenophen-2-yl)stibine

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Abstract

A new tertiary stibine and bismuthine containing C-heterocyclic selenophene ring have been synthesized. Tris(2-selenophenyl)stibine (**1**) and tris(2-selenophenyl) bismuthine (**2**) and characterized by IR, MS, ^{1}H, ^{13}C, COSY, HETCOR NMR spectroscopy. Crystal structures of stibine and bismuthine have been determined. In an attempted synthesis of tris(2-selenophenyl) phosphine, an oxidative product tris(2-selenophenyl) phosphine oxide (**3**) was isolated under the experimental conditions used, whose X-ray crystal structure was also determined. To best of our knowledge C-heterocyclic selenophene-2-yl pnictogens are first of their kind, as not many examples are known even with other p-block metal/metalloids.

The biological activity of compound (**1**) was determined. The compound (**1**) shows a significant selectivity (>85%) for carcinogenic cell K and U growth inhibition. The toxicity of tris(selenophen-2-yl) stibine (**1**) on larvae of Artemia Salina was studied and the LC_{50} value was 589.6 μM.

Keywords: Selenophene; Heterocyclic stibine; Bismuthine; Sb–Se intermolecular intractions; Biological activity

1. Introduction

Tertiary stibines R_3Sb or bismuthines R_3Bi (where R = alkyl or aryl) are very well known but there exist only few reports on tertiary stibines or bismuthines where antimony or bismuth atom is directly attached to an aromatic heterocycle, respectively [1–4]. Recently our group have reported some new stibines and bismuthines containing C-attached 2-thienyl, 2-furyl and 2-(*N*-methylpyrrolyl) group. X-ray structures and biological activities of these stibines and bismuthines have also been reported [5–7]. On the other hand, selenophen-2-yl substituted derivative of p-block metals/metalloids are very rare in literature, which is evident from the fact that a framework (including any metal-selenophenyl, benzoselenophenyl and their η-complexes) generates only 97 entries through a search of the Science Finder. Only a very few organometallic compounds of silicon and tin are known with selenophen-2-yl group [8–12]. In 2003, the very first crystal structure of $Hg(C_4H_3Se)_2$ containing C-heterocyclic selenophen-2-yl ring, has appeared [13]. There exist reports where selenophene derivatives were used for non linear optical (NLO) properties [14–18]. In addition, biological applications of organoselenium compounds are widely known [19–21]. There are very few reports in the literature on stibines or bismuthines containing C-heterocyclic rings and no reports on these compounds containing C-heterocyclic selenophene. Because of our

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interest in antimony ligands and pnictogen chalcogenide thin films [5,22–25], this work was undertaken.

2. Results and discussion

In this paper, we describe the synthesis of tris(selenophen-2-yl)stibine (**1**) and tris(selenophen-2-yl) bismuthine (**2**) and tris(selenophen-2-yl) phosphine oxide (**3**) by the reaction of (selenophen-2-yl)lithium with corresponding trihalides. Stibine and bismuthine remain unaffected by water thus Sb–C and Bi–C bonds in these compounds are not hydrolyzed by water alone. The compounds (**1**) and (**2**) show a slow degree of decomposition at room temperature in moist atmosphere. The decomposition products could not be characterized because of its insolubility in common organic solvents. In comparison to stibine and bismuthine, tris(selenophen-2-yl)phosphine is sensitive to air and forms oxide. Compounds (**1**) and (**2**) are soluble in $CHCl_3$, THF and show a little solubility in pentane and hexane, while compound (**3**) is completely insoluble in hexane and pentane.

The principal band observed in UV spectra of these stibines and bismuthines can be assigned to a π–π* transition in the heterocycle. EI mass spectral analyses of stibine and phosphine oxide show molecular ion peaks at 511 and 437 *m*/*z* confirming the molecular formula. In the case of bismuthine molecular ion peak could not be observed in EI spectrum while in FAB^+ spectrum, molecular ion peak at 599 *m*/*z* was observed which confirm the molecular formula. The fragmentation peaks observed for the hetero cyclic part are not of much importance and are according to those reported in the literature.

The proton NMR spectra of all these compounds show a characteristic pattern of three multiplets in the aromatic region [26,27]. In all the cases, assigning individual protonic signal was based on J_{HH} coupling constant values and was confirmed by COSY. ^{31}P NMR chemical shifts of (**3**) ($\delta = 21.8$) show an upfield shift in comparison to $Ph_3P{=}O$ ($\delta = 29.3$) which is because of less aromatic and sterically demanding (bulky) character of selenophene ring.

At ambient temperature, four ^{13}C NMR signals were observed for these compounds in the aromatic region.

X-ray diffraction parameters, selected bond lengths and bond angles for compound (**1**), (**2**) and (**3**) are listed in Tables 1 and 2, respectively. The molecular structures of (**1**), (**2**) and (**3**) are shown in Fig. 1–3, respectively.

On the basis of the structures of other tertiary stibines and bismuthines containing *C*-heterocyclic ring [5,6], a pyramidal structure may be assigned to (**1**) and (**2**). Stibine $(C_4H_3Se)_3Sb$ (**1**) is pyramidal. The structure shows that it can act as a tripodal ligand.

The average Sb–C bond length found in **1** is 2.127 Å, which is slightly shorter than that found in other known tertiary stibines [28–31], e.g., Ph_3Sb 2.155; 2.190 in tris(2,6-dimethylphenyl)stibine; 2.158 in tris(2-methylphenyl)stibine. This may be due to the pπ–dπ* bonding to a greater extent and thus shortening of Sb–C bond length and is similar to $(C_4H_3S)_3Sb$ where $C–Sb_{(av)}$ is 2.129 Å [5].

For the crystal structures of (**1**) and (**2**) an electron-density map phased by direct methods revealed the essential features of the structures. However, attempts to improve the positions of the ring atoms in one of the selenophene rings with a series of difference Fourier syntheses proved unsuccessful. Each successive map

Table 1
Crystal data for compounds (**1**), (**2**) and (**3**)

Compounds	(**1**)	(**2**)	(**3**)
Empirical formula	$C_{12}H_9Se_3Sb$	$C_{12}H_9Se_3Bi$	$C_{12}H_9Se_3PO$
Formula weight	511.82	599.05	437.04
Crystal system	Triclinic	Monoclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/c$	$P2_1/c$
Crystal size	$0.252 \times 0.236 \times 0.044$	$0.60 \times 0.24 \times 0.14$	$0.364 \times 0.304 \times 0.026$
a (Å)	6.3316(4)	14.588(1)	8.2653(7)
b (Å)	8.411(1)	10.0894(7)	16.643(1)
c (Å)	13.848(1)	19.411(1)	10.4100(9)
α (°)	77.198	90	90
β (°)	80.286	93.507(2)	99.461(2)
γ (°)	83.165	90	90
Volume ($Å^3$)	706.3(1)	2851.6(3)	1412.5(2)
Z	2	8	4
Density (calc) (Mg/cm^3)	2.407	2.791	2.055
μ (mm^{-1})	9.655	19.995	7.905
Reflections collected	8454	16432	19233
Independent reflections	2485	5008	5109
R	0.0709	0.0432	0.0455
GOF	1.040	0.962	0.892
Δ/σ (e $Å^{-3}$)	5.269/1.967	1.079//1.449	1.183/−0.637

Table 2
Selected bond lengths (Å) and bond angles (°)

Compound **1**			
Sb(1)–C(15)	2.143(12)	Sb(1)–C(10)	2.122(11)
Sb(1)–C(5)	2.117(11)	Se(1A)–C(5)	1.797(12)
Se(1A)–C(2A)	1.86(4)	Se(6)–C(7)	1.828(2)
Se(6)–C(10)	1.855(11)	Se(1B)–C(5)	1.832(10)
Se(11)–C(12)	1.836(14)	Se(11)–C(15)	1.872(11)
C(5)–Sb(1)–C(15)	96.1(4)	C(10)–Sb(1)–C(15)	94.0(4)
C(5)–Sb(1)–C(10)	97.3(4)	C(5)–Se(1A)–C(2A)	89.3(1)
C(5)–Se(1B)–C(2B)	87.3(10)	C(12)–Se(11)–C(15)	87.0(6)
Compound **2**			
Bi(1)–C(2)	2.216(12)	Sb(2)–C(22)	2.187(12)
Bi(1)–C(7)	2.238(11)	Bi(2)–C(17)	2.210(12)
Bi(1)–C(12)	2.239(12)	Bi(2)–C(27)	2.242(11)
Se(6)–C(10)	1.855(11)	Se(1B)–C(5)	1.832(10)
Se(11)–C(12)	1.836(14)	Se(11)–C(15)	1.872(11)
C(2)–Bi(1)–C(7)	95.6(4)	C(2)–Bi(1)–C(12)	93.1(4)
C(7)–Bi(1)–C(12)	92.1(4)	C(2)–Bi(1)–Se(5)	150.6(3)
C(7)–Bi(1)–Se(5)	76.8(3)	C(12)–Bi(1)–Se(5)	115.3(3)
Compound **3**			
P(1)–O(1)	1.478	P(1)–C(12)	1.766(4)
P(1)–C(7)	1.785(3)	P(1)–C(2)	1.784(3)
O(1)–P(1)–C(12)	113.86(2)	O(1)–P(1)–C(2)	113.29(14)
C(2)–P(1)–C(12)	104.80(2)	O(1)–P(1)–C(7)	113.99(2)

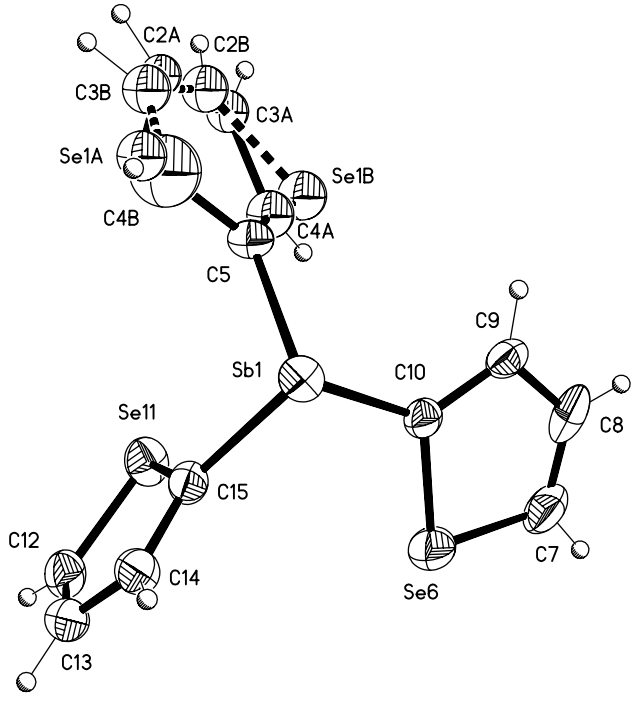

Fig. 1. ORTEP-like view of tris(selenophen-2-yl)-stibine (**1**) showing the orientational disorder. Anisotropic displacement parameters at 30% probability level.

gave consistently unrealistic bond distances within the selenophene ring, which suggested the presence of disorder. An isotropic full-matrix least-squares calculation at this juncture verified the existence of disorder in the structure. In particular the C_{ipso}–Se and C_{ipso}–C_β distances both refined to 1.69 Å while the temperature factors for C_β and selenium became negative and unreasonably large, respectively. This result is what may be expected for a disordered structure.

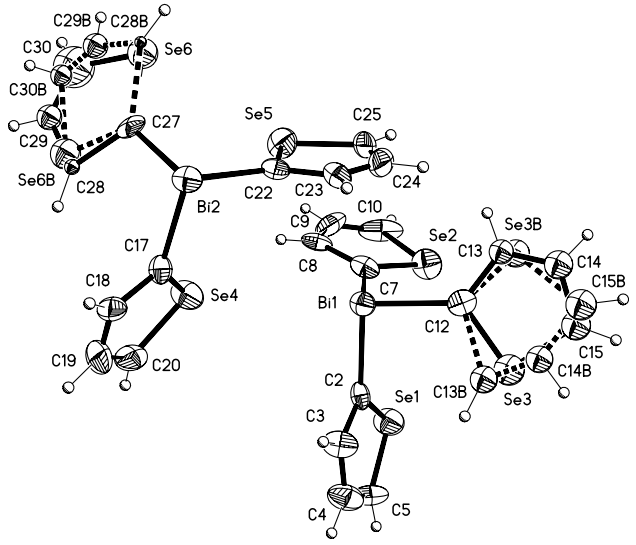

Fig. 2. ORTEP-like view of tris(selenophen-2-yl)-bismuthine showing the orientational disorder. Anisotropic displacement parameters at 30% probability level.

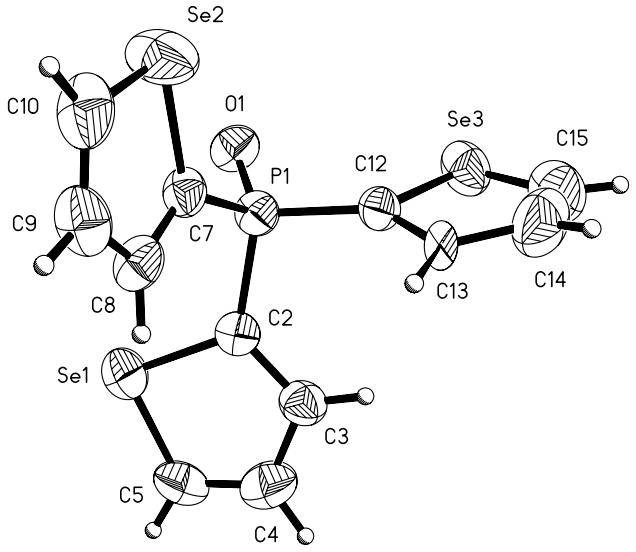

Fig. 3. Molecular structure of compound (**3**).

The disorder is twofold in each case, and superimposes the β and β′ Se- and C-atom sites. The populations of Se and C scattering factors were refined for each of these sites, with the usual constraints – that the total population each site be 1.0, and that each selenophene rings have the correct overall stoichiometry – that is, C_4H_3Se. Each site was refined with its own set of anisotropic displacement parameters (except for the minor component Se3B, C13B, C14B C15B of one of the two independent molecules of **2**), in order that the observed

scattering density be modeled as accurately as possible. Figs. 1 and 2 show the structures of the molecules, with the atomic sites labeled according to one of the disordered congeners.

The rotation of the selenophene rings about the M–C_{ispo} bond (α angle) is surprisingly similar in both series of compounds considering the range of possible conformations. This fact indicates that the intramolecular interactions between the selenophene rings are quite comparable in both structures. The values of α in the stibine are: 83.6°, 53.5°, 74.9° while in the bismuthine are: 72.1°, 72.4°, 64.6° and 65.8°, 74.1°, 51.8° for the two independent molecules in the asymmetric unit, respectively. The difference in the crystal structures and packing must then involve the variation of the intermolecular contacts as a function of the particular central atom. This conclusion is supported by observation that intermolecular contacts are completely different (see Table 3) in the two series of compounds.

An examination of the intra and intermolecular contacts, strongly suggests that the observed disorder is the result of an alternation of a helical arrangement of the selenophene rings for the "isolated" molecules which minimize the Se–Se intramolecular interactions, while a non-helical arrangement with strong M–Se intermolecular interactions arise for the crystal structure.

For the stibine, a penta coordinated polymeric structure along the ***a*** direction is observed (Fig. 4) via centro- and non-centrosymmetric Se–Sb interactions just of the disordered selenophene ring. For the bismuthine (Fig. 5) again, a polymeric structure along the ***a*** direction is observed, but now the central Bi-atom is six-coordinated involving centro- and non-centrosymmetric Se–Bi interactions of the ordered and disordered selenophenyl rings alternating the two crystallographic independent molecules. Taking in account these Sb–Se and Bi–Se interactions compound (**1**) is trigonal bipyramidal and compound (**2**) is trigonal antiprism, respectively.

The geometry around phosphorus in tris(selenophen-2-yl) phosphine oxide (**3**), is approximately tetrahedral. The P–O bond length (1.478(2) Å) is shorter than the P–O bond lengths in various polymorphs of Ph_3PO (1.484(1) Å which may be due to the enhanced electron withdrawing character of selenophene ring the case of (**3**).

Table 3
Intra- and inter-molecular Se···M interactions

Sb1···Se1B_(1 + *x*, *y*, *z*)	3.902(3)
Sb1···Se1A_(1 − *x*, −*y*, −*z*)	3.796(5)
Bi1···Se4	3.980(2)
Bi1···Se5	4.052(2)
Bi1···Se3_(2 − *x*,1 − *y*, 2 − *z*)	3.906(2)
Bi2···Se1_(*x*, 1 + *y*, *z*)	4.063(2)
Bi2···Se2_(*x*, 1 + *y*, *z*)	4.206(2)
Bi2···Se6B_(1 − *x*, 2 − *y*, 2 − *z*)	3.765(5)

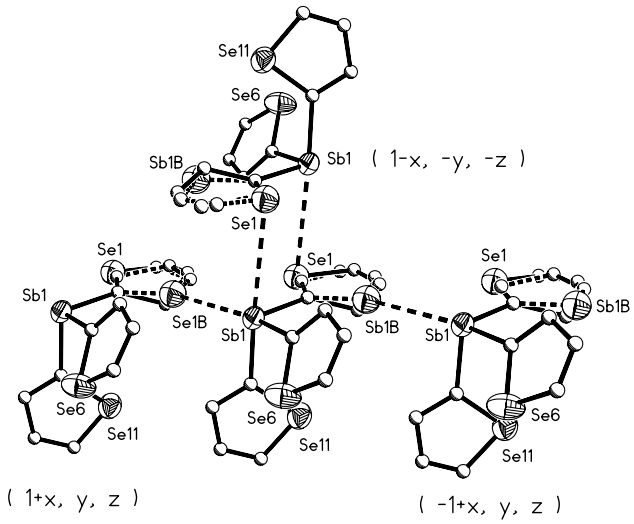

Fig. 4. Crystal packing of tris(selenophen-2-yl)-stibine showing the Se···Sb contacts. Anisotropic displacement parameters at 30% probability level.

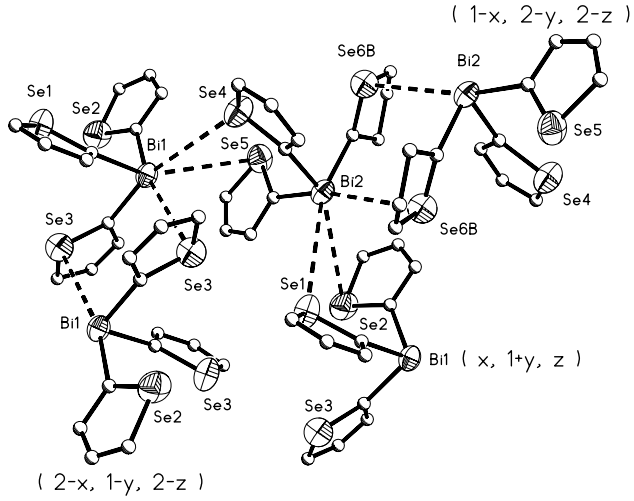

Fig. 5. Crystal packing of tris(selenophen-2-yl)-bismuthine showing the Se···Bi contacts. Anisotropic displacement parameters at 30% probability level.

In phosphine oxides, the bond order of the P–O bond has been a matter of considerable debate which estimates ranging from ~1.0 to 3.0. There are at least two methods available for estimation of bond order from empirical data: the bond distance-bond order (BDBO) relationship and IR force constants. In materials science, BDBO relationship has been further refined as Bond Valence Model [32–35]. The central equation used here for the BDBO relationship is

$$BO_{po} = \exp[(R_{po} - d_{po})/0.37],$$

where BO_{po} is the bond order between P and O atoms, d_{po} is the observed bond distance, and R_{po} is the single bond expectation distance, which can be calculated as 1.7 Å for P–O bond. The value 0.37 (sometimes called

the universal constant) is taken from a number of recent publications [32–36]. The Bond order calculated is 1.80 for P–O bond in compound (3) from this method. Calculation of bond order from the IR force constants can be done using P–O stretching frequency, which is found at 1192 cm^{-1} in (**3**), gives a value of 1.77. It is reported in literature that greater electron withdrawing character would lead to higher bond order. The estimated bond order is little higher than Ph_3PO and very similar to $(p\text{-ClPh})_3PO$, showing the electron withdrawing character of selenophene-2-yl ring [36].

The toxicity of tris(selenophen-2-yl) stibine on larvae of Artemia Salina, LC_{50} was determined. The LC_{50} value was 589.6 μM shows that this is more toxic than tris(2-thienyl)stibine (LC_{50} = 29.5 μM).

The compound (**1**) shows a significant selectivity (>95%) for carcinogenic cell K562 and U251 growth inhibition and show a less inhibition for HCT15 (>40%). But this compound is highly toxic for normal lymphocytes con ~ 75% lethality.

In conclusion, very new selenophen-2-yl pnictogen derivatives have been synthesized and characterized. X-ray structures of all the compounds have also been determined. The ligating behavior of these compounds with Pt, Pd and Rh and their possible application in catalysis is under progress.

3. Experimental

All the solvents were distilled immediately prior to use. All the reactions were performed under an atmosphere of oxygen-free, dry nitrogen. Elemental analyses were determined on a Perkin–Elmer 240. Melting points were obtained on a MEL-TEMP II Fisher and are uncorrected. EI and FAB^+ mass spectra were recorded on a JEOL SX102 double-focusing mass spectrometer with reverse geometry using a 6-kV Xenon beam (10 am); nitrobenzyl alcohol was used as matrix for recording the mass spectra. IR spectra were recorded on a Nicolet-Magna 750 FT-IR spectrometer as nujol mulls. ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ or CD_3OD on JEOL ECLIPSE 300 (^1H: 300.5311 MHz; ^{13}C: 75.5757 $^{31}P\{^1H\}$: 121 MHz) spectrometer. The cytotoxicity experiments against carcinogenic cells [37] leukaemia K562, CNS U251 and colon cancer HCT15 and toxicity experiments against Artemia Salina [6] were carried out as reported earlier by our group.

3.1. X-ray crystallography

The X-ray intensity data were measured at 293 K on a Bruker SMART APEX CCD-based X-ray diffractometer using a monochromatized Mo Kα radiation (λ 0.71073 Å). The detector was placed at a distance of 4.837 cm from the crystals in all cases. Analysis of the data showed in all cases negligible decays during data collections. An analytical face indexed absorption correction was applied. Crystal structures were refined by full-matrix least squares. SMART software (data collection and data reduction) and SHELXTL were used for solution and refinement of the structures.

3.2. Tris(selenophen-2-yl)stibine (*1*)

A solution of antimony trichloride (0.485 g, 2.13 mmol) in ether (10 ml) was added drop-wise and under a nitrogen atmosphere to (2-selenophene-2-yl)lithium which was synthesized "in situ" according to reported method [38] (a solution of 0.212 mL of selenophene in 20 mL of THF and 4 mL of ^{n}BuLi (1.6 M in hexane) under inert atmosphere at −78 °C for 45 min) in THF at −20 °C with continuous stirring. The mixture was further stirred for 3 h at room temperature (r.t.) and then reaction was quenched with ice. After extraction with dichloromethane (3 × 10 ml) and drying over sodium sulfate, solvent was removed under vacuum. Slow concentration from chloroform solution afforded single crystals suitable for X-ray analysis. Yield: 0.92 g (84%); m.p.: 39–40 °C; IR (ν cm^{-1}): 476 (Sb–C), 3081 (C–H aromatic); ^1H NMR ($CDCl_3$, δ in ppm): 7.39 (d, 1H, H_3, J_{34} = 3.84), 7.23 (1H, H_4) 8.83 (1H, H_5, J_{45} = 5.90), ^{13}C NMR ($CDCl_3$, δ in ppm): 139.93 (C_3), 129.74 (C_2), 137.76 (C_5), and 130.98 (C_4). MS (EI) *m/z* (%): MS (EI) *m/z* (%): 511(4) [M^+], 381(3) [$M - (C_4H_3Se)^+$], 251(100) [$M - 2(C_4H_3Se)^+$], 262(29) [$(C_4H_3Se)2$].

3.3. Tris(selenophen-2-yl)bismuthine (*2*)

The compound was synthesized by a similar procedure to (**1**). $BiCl_3$ (0.68 g, 2.13 mmol) was dissolved in THF (10 mL). Single crystals suitable for X-ray analysis were obtained from dichloromethane–pentane solution. Yield: 0.80 g (62%); m.p.: 52 °C; IR (ν cm^{-1}): 481(Bi–C), 3079 (C–H aromatic); ^1H NMR ($CDCl_3$, δ in ppm): 8.40 (d, 1H J_{45} = 5.85, H_5), 7.78 (1H, H_3), 7.36 (1H, H_4 J_{34} = 3.8). ^{13}C NMR ($CDCl_3$, δ in ppm): 131.99 (C_4), 138.13 (C_3), 140.61 (C_5), and 129.51 (C_2). MS (FAB^+) *m/z* (%): 601(4) [M^+], 470(10) [$M - (C_4H_3Se)^+$], 340(12′) [$M - 2(C_4H_3Se)^+$], 262(29) [$(C_4H_3Se)2$].

3.4. Tris(selenophen-2-yl)phoshine oxide (*3*)

The compound was synthesized by a similar procedure to (**1**). PCl_3 (0.291 g, 2.13 mmol) was dissolved in THF (10 mL). During work up it gets oxidized. Single crystals suitable for X-ray analysis were obtained from dichloromethane-pentane solution at −30 °C. Yield: 0.82 g (88%); m.p. 88 °C; IR (ν cm^{-1}): 471 (P–C), 3090 (C–H aromatic), 1192 (P–O), ^1H NMR ($CDCl_3$, δ in ppm): 7.83 (d, 1H, H_3), 7.44 (1H, H_4, J_{45} = 4.90),

8.49 (1H, H_5). ^{13}C NMR ($CDCl_3$, δ in ppm): 130.77 (C_4), 139.01 (C_3), 140.61 (C_5), and 122.85 (C_2). MS (EI) *m/z* (%): 437(56) [M^+], 309 (12) [$M - (C_4H_3Se)^+$], 293(100) [$M - 2(C_4H_3Se)O^+$], 262(30) [$2(C_4H_3Se)$].

4. Supplementary materials

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 263754–263756 for compounds **1**–**3**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ UK [Fax. (int code) +44(1223)336-033, or Email: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk.

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