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Article

The X-Ray Crystal Structures of Primary Aryl Substituted Selenoamides

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Abstract: The X-ray structures of 12 primary selenoamides are reported. Metric parameters are provided, together with an illustration of the range of hydrogen bonding motifs.

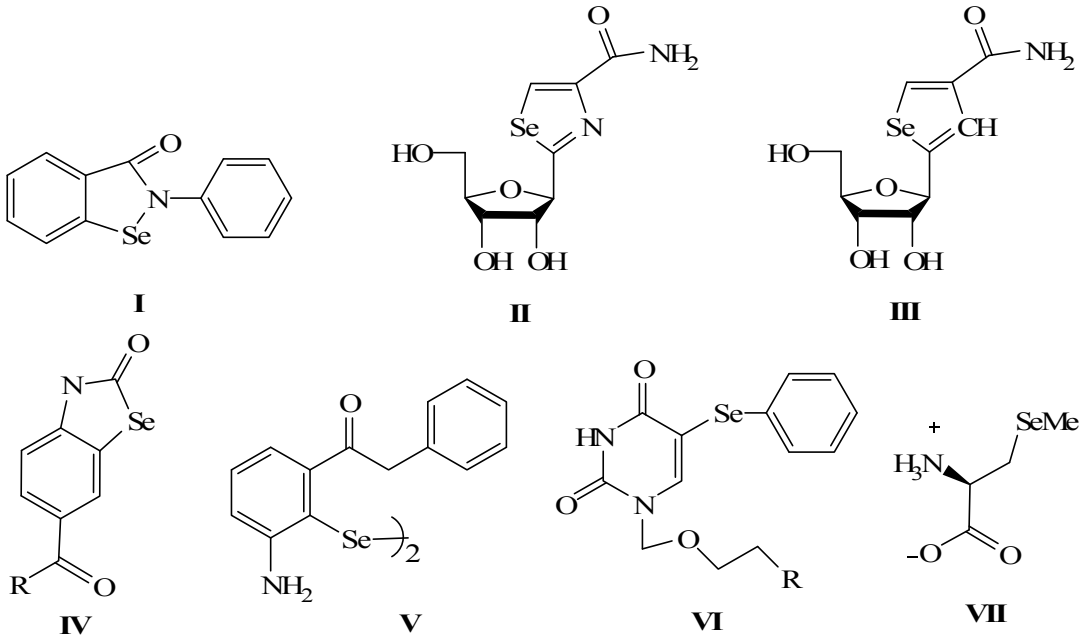
Keywords: Woollins reagent; Primary selenoamides; X-ray structures.

Introduction

Selenium is an essential element for life; e.g, selenocysteine is recognised as the 21st amino acid and the importance of selenium containing enzymes in redox processes has is now recognized [1-3]. Many organoselenium compounds have been studied as biological models that simulate catalytic functions demonstrated by natural enzymes [4-14]. For example, ebselen (**I**, Figure 1) acts as a glutathione peroxidase (GP_x) mimic and as a scavenger of peroxinitrite and the activity of the sulfur analogue of ebselen was 15-fold lower than that of ebselen [5,6]. Selenazofurin (**II**) has been reported to be a potent inhibitor of phlebovirus infections [10]. Selenophenfurin (**III**) exhibits antiproliferative and inosine 5'-monophosphate dehydrogenase (IMPDH)-inhibition activity. Leukotrienes such as leukotriene B_4 (LTB_4) are important mediators of asthma, allergy, arthritis, psoriasis, and inflammatory bowel disease [11,12]. Galet *et al.* showed that benzoselenazolinones of type **IV** and the corresponding diselenides **V** dramatically decrease the formation of LTB_4 [13]. Phenylseleno-substituted pyrimidines of type **VI** exhibit significant inhibitory properties on Urd Pase and TMS. Se-

methyl selenocysteine (**VII**) was found to be an antitumor agent, and it has been shown that *β*-elimination reaction is important for this activity [14].

Figure 1. Some biologically active selenium compounds.



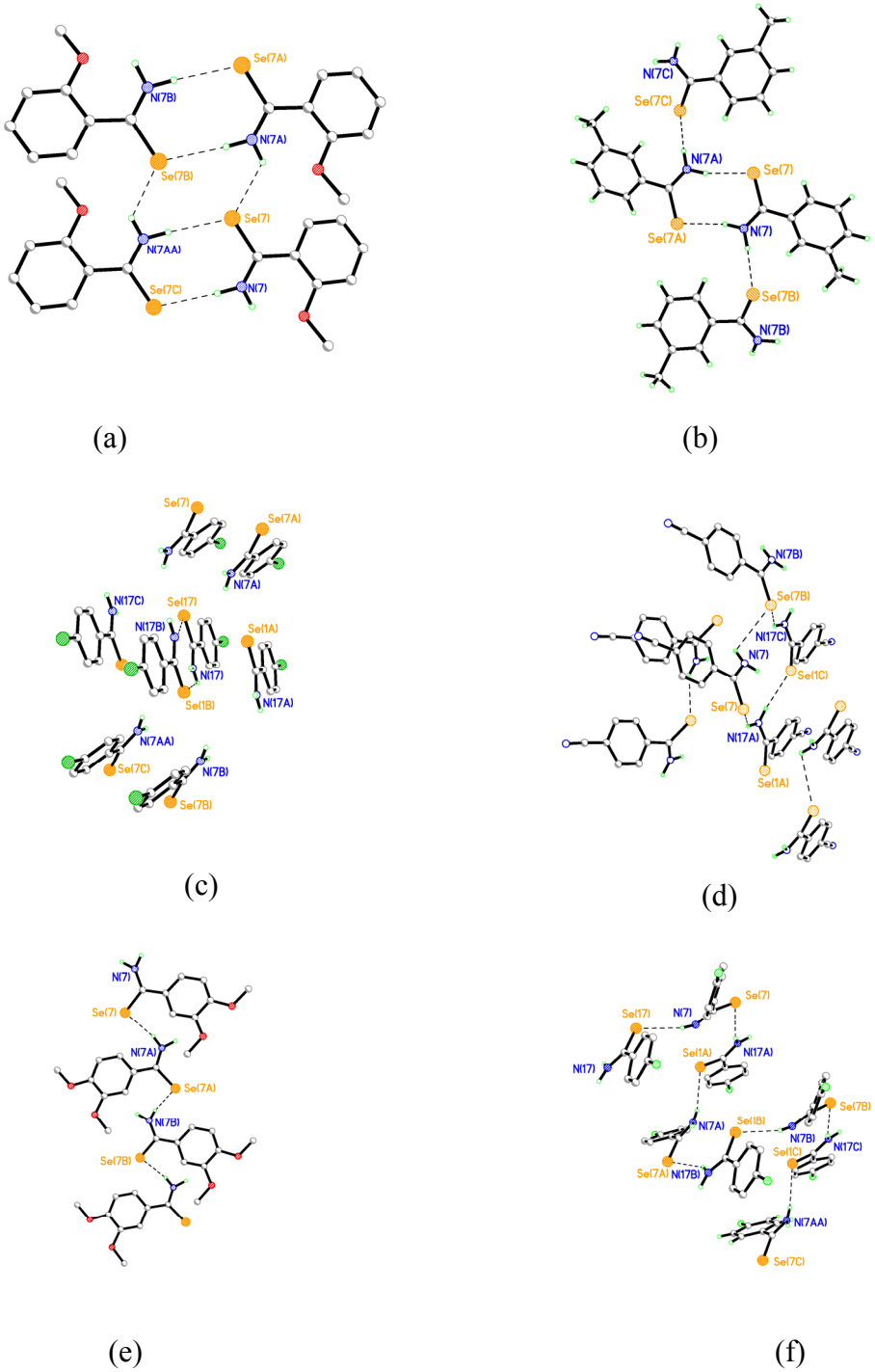
We have a long term interest in selenium chemistry [15-20]. Woollins reagent (**WR**, 2,4-bis(phenyl)-1,3-diselenadiphosphetane-2,4-diselenide, $[PhP(Se)(\mu\text{-}Se)]_2$), which is isostructural with the thionation agent $[(p\text{-}MeOC_6H_4)P(S)(\mu\text{-}S)]_2$ (Lawesson's reagent), and may be obtained readily from $PhPCl_2$, Na_2Se and Se [21], is an excellent selenation reagent for converting a range of unsaturated organic substrates into unusual phosphorus containing heterocycles [22-31]. We have recently reported the use of **WR** for organic transformations and for the facile synthesis of primary arylselenoamides from **WR** and ArCN [32]. Although the X-ray crystal structures of some tertiary and secondary selenoamides have been documented [33-39], surprisingly, no structural information has been published on primary arylselenoamides $ArC(Se)NH_2$. We here provide a comparative study of a range of these systems.

Results and Discussion

Selected metric parameters for compounds **A** - **L** are given in Table 1. The C=Se bond lengths range from 1.822(5) to 1.856(4) Å whilst the C-N bond lengths are in the range 1.270(7) to 1.324(8) Å. This compares with literature values from the Cambridge Database for amide C-Se distances of 1.787-1.885 and C-N of 1.29-1.34 Å. The amide functionality is not particularly coplanar with the aryl backbone, with the selenium atom lying up to 1.406 Å from the aryl ring mean plane with the C(7)-N(7)-Se(7) plane being up to 87° from the aryl mean plane in this case. This maximum deviation from coplanarity may be a function of the presence of two ortho chlorine substituents in compound **L** causing repulsion. However, it is interesting to note that in compound **F** the two independent

molecules have quite different degrees of coplanarity for the selenoamide functional group and the aryl ring suggesting that there is little electronic reason for coplanarity.

Figure 2. Examples of H-bonding motifs. (a) **1** ladder (b) **2** linked dimer (c) **7** Herring-bone dimers (d) **6** tetrameric sheets (e) **10** Chains (f) **9** Helical Chains.



Although hydrogen bonding is well understood for N-H···O and N-H···S systems, there is less data available for N-H···Se systems. We have previously noted that $Ph_2P(Se)NHP(Se)Ph_2$ forms dimers via N-H···Se hydrogen bonds (Se···N 3.19, Se···H 2.52 Å) [40] and it is interesting to note the range of

motifs that we have observed in compounds **A** – **L** (Figure 2). We have broadly classified the pattern of hydrogen bonding in compounds **A** - **L** and give selected parameters in Table 2 and provide illustrative examples in Figure 1. It is clear that N-H···Se hydrogen bonding is an important feature of the solid state packing of these molecules and may be a significant influence in biological systems.

Table 1. Selected ^{77}Se NMR data, bond lengths (Å) and angles (^{o}) (Rows containing multiple entries are a.consequence of the presence of more than one independent molecule in the asymmetric unit).

	δ_{Se} (ppm*)	C=Se (Å)	C-N (Å)	Aryl/Scene interplanar angle (°)	Deviation of Se from aryl Mean plane (°)
A	602.1	1.856(4)	1.311(5)	21	0.469
B	641.2	1.848(3)	1.314(3)	36	1.118
C	628.6	1.829(7)	1.318(9)	27	0.619
D	608.7	1.843(5)	1.316(7)	14	0.343
E	579.5	1.848(5)	1.317(6)	17	0.576
F	703.7	1.840(11)	1.292(15)	7	0.157
		1.846(14)	1.332(17)	40	1.087
G	646.5	1.855(10)	1.310(13)	10	0.173
		1.844(12)	1.295(17)	3	0.104
H*	647.2	1.81(5)	1.27(7)	8-32	0.299 [-0.963]
I	629.6	1.829(6)	1.324(8)	37 [18]	1.018 [0.361]
		[1.838(6)]	[1.305(8)]		
J	529.0	1.829(6)	1.324(10)	39	0.786
K	649.9	1.838(3)	1.317(4)	48	1.346
L	715.8	1.822(5)	1.298(7)	87	1.406

*Five independent molecules in the asymmetric unit, average bond lengths and ranges of Se deviations/interplanar angles are given.

Table 2. Major N-H···Se hydrogen bonding distances (Å).

	Type	Se···H	Se···N	Se···H-N	Se···H	Se···N	Se···H-N
A	Ladder	2.55(1)	3.512(3)	167(3)	2.72(4)	3.403(3)	127(3)
B	Linked dimers	2.527(7)	3.489(2)	168(2)	2.539(10)	3.491(2)	164(3)
C	Linked dimers	2.59(3)	3.510(6)	156(6)	2.58(3)	3.491(5)	155(5)
D	Linked dimers (sheets)	2.55(1)	3.517(4)	170(5)	2.71(5)	3.408(4)	129(4)
E	Linked dimers (sheets)	2.55(7)	3.527(4)	174(4)	2.82(5)	3.415(4)	120(4)
F	Tetramers (sheets)	2.57(2)	3.58(11)	171(11)	2.63(6)	3.527(11)	152(11)
		2.90(14)	3.430(12)	115(1)	2.69(10)	3.466(10)	136(10)
G	Herringbone dimers	2.68(10)	3.502(8)	142(12)	2.85(12)	3.509(8)	125(1)
H	Dimers	2.50(17)	3.43(4)	158(4)	2.49(16)	3.43(3)	160(4)
		2.48(9)	3.45(5)	169(5)	2.58(8)	3.43(3)	169(5)

Table 2. *Cont.*

I	Helical chain	2.63(3)	3.513(5)	150(4)	2.97(3)	3.628(5)	125(4)
		2.74(4)	3.566(5)	143(5)	2.62(3)	3.512(5)	151(5)
J	Chain	2.52(2)	3.483(6)	168(6)			
K	Dimers	2.69(1)	3.63(3)	162(3)			
L	Linked dimers	2.535(8)	3.511(4)	174(5)	2.579(14)	3.533(4)	165(4)

N-H···O hydrogen bonding: [a] H···O 2.09(4) Å, N…O 3.016(7) Å, N-H…O 156(7) Å; [b]H···O 2.32(3) Å, 2.24(3) Å; N···O 3.076(4), 2.923(3) Å; N-H···O 133(3), 126(3) Å.

Experimental

Primary arylselenoamides **A** – **L** (Figure 3) were prepared as described previously [32]. Their X-ray crystal data (Table 3) were collected at 93 K by using a Rigaku MM007 High brilliance RA generator/confocal optics and Mercury CCD system. Intensities were corrected for Lorentz-polarisation and for absorption. The structures were solved by direct methods. Hydrogen atoms bound to carbon were idealised. Structural refinements were obtained with full-matrix least-squares based on F^2 by using the program SHELXTL [41]. CCDC 611494 & 611495 CCDC 713559 - 713568 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via 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; E-mail: deposit@ccdc.cam.ac.uk.

Figure 3. The chemical structures of primary arylselenoamides **A** – **L.**

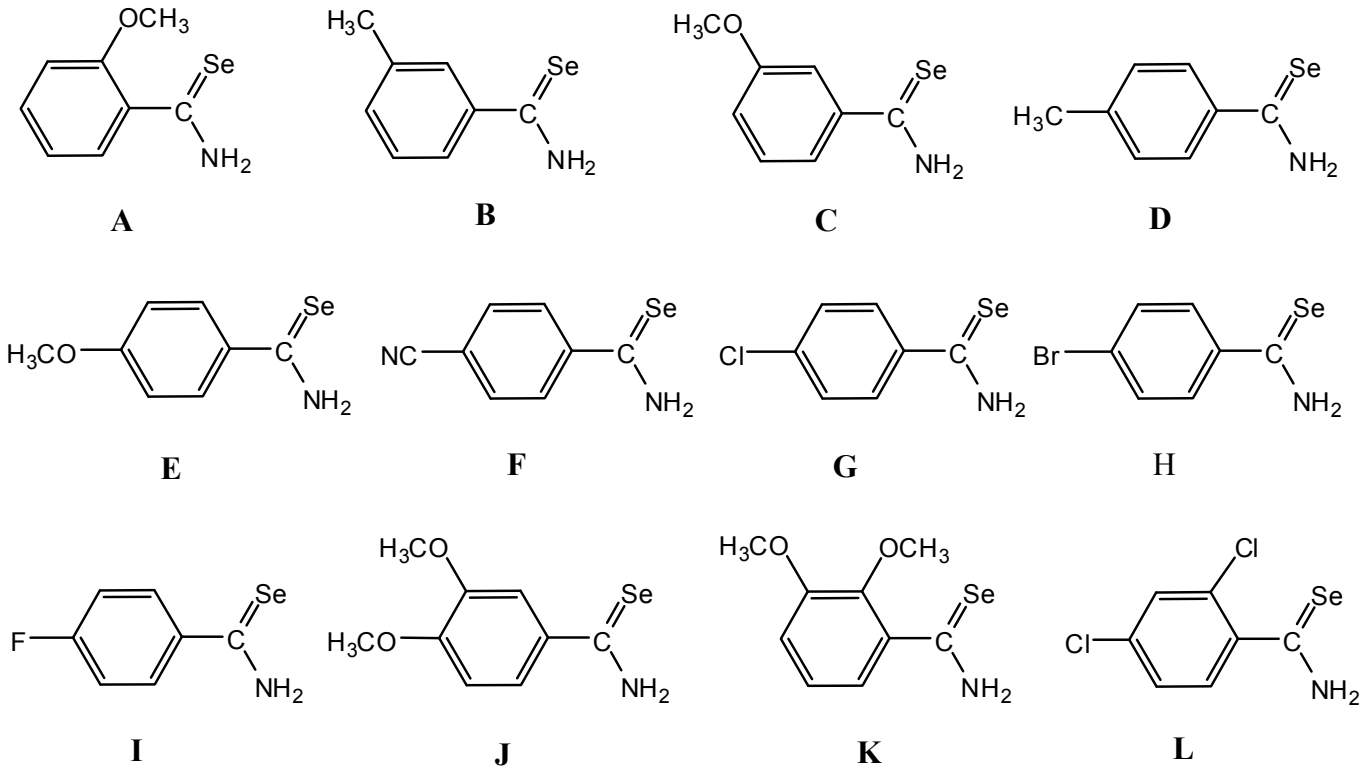


Table 3. Details of data collections and refinements for **A** - **L**.

Compound	**A**	**B**	**C**	**D**	**E**	**F**
Empirical formula	C_8H_9NOSe	C_8H_9NSe	C_8H_9NOSe	C_8H_9NSe	C_8H_9NOSe	$C_8H_6N_2Se$
Crystal color, habit	Yellow, prism	Orange, needle	Orange, prism	Orange, needle	Yellow, platelet	Orange, needle
Crystal dimensions/mm	0.20 × 0.15 × 0.05	0.15 × 0.15 × 0.08	0.20 × 0.20 × 0.10	0.25 × 0.05 × 0.01	0.20 × 0.05 × 0.02	0.30 × 0.06 × 0.03
Crystal system	Monoclinic	Monoclinic	Orthorhombic	Monoclinic	Monoclinic	Orthorhombic
Space group	P2(1) / c	P21(1) / c	Pbca	P21(1) / c	P2(1) / c	P2(1)2(1)2(1)
a	5.9600(11)	7.5986(15)	8.4086(17)	9.869(2)	10.108(4)	7.4345(15)
b	9.9600(18)	10.464(2)	11.586(2)	6.0039(13)	6.016(2)	6.0647(12)
c	14.114(3)	10.163(2)	16.960(4)	13.658(3)	13.765(6)	34.203(6)
β	95.506(5)	96.303(6)		105.485(6)	106.907(13)	
U/ Å^3	833.9(3)	803.2(3)	1652.3(6)	779.9(3)	800.9(6)	1542.1(5)
Z	4	4	8	4	4	8
M	214.1	198.1	214.1	198.1	214.1	209.1
Dc/g cm^{-3}	1.705	1.638	1.722	1.687	1.776	1.801
μ/mm^{-1}	4.441	4.595	4.483	4.732	4.625	4.796
F(000)	424	392	848	392	424	816
Measured reflections	4447	4308	6679	2899	4170	8341
Independent reflections (R_{int})	1498 (0.0393)	1502 (0.0396)	1377 (0.0908)	1057 (0.0393)	1385 (0.0629)	2715 (0.0562)
Final R1, wR2[I>2σ(I)]	0.0316, 0.0971	0.0269, 0.0680	0.0604, 0.1252	0.0371, 0.0924	0.0458, 0.0911	0.0752, 0.1501
Compound	**G**	**H**	**I**	**J**	**K**	**L**
Empirical formula	C_7H_6ClNSe	C_7H_6BrNSe	C_7H_6FNSe	$C_9H_{11}NO_2Se$	$C_9H_{11}NO_2Se$	$C_9H_{11}NO_2Se$
Crystal color, habit	Orange, prism	Orange, prism	Yellow, prism	Yellow, patelet	Yellow, prism	Yellow, patelet
Crystal dimentions/mm	0.30 × 0.20 × 0.05	0.10 × 0.08 × 0.05	0.30 × 0.20 × 0.10	0.20 × 0.20 × 0.05	0.20 × 0.20 × 0.15	0.20 × 0.20 × 0.05
Crystal system	Triclinic	Triclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	P-1	P-1	C2 / c	Cc	P2(1) /n	Cc
a	4.0219(8)	12.532(3)	31.874(4)	9.905(2)	10.7387(17)	9.905(2)
b	10.774(2)	12.720(3)	3.9871(6)	14.311(3)	7.0242(11)	14.311(3)
c	17.939(4)	17.285(6)	22.322(3)	7.1924(16)	13.539(2)	7.1924(16)
α	92.421(6)	101.69(2)	90	90	90	90
β	92.548(6)	99.83(2)	97.724	104.360(6)	106.225(4)	104.360(6)
γ	91.464(6)	113.572(17)	90	90	90	90
U/ Å^3	775.6(3)	2373.8(11)	2811.1(7)	987.7(4)	980.6(3)	987.7(4)
Z	4	12	16	4	4	4
M	218.5	263.00	202.1	244.1	244.1	244.1
Dc/g cm^{-3}	1.872	2.208	1.910	1.642	1.654	1.642
μ/mm^{-1}	5.102	9.713	5.274	3.768	3.796	3.768
F(000)	424	1488	1568	488	488	488
Measured reflections	2011	4531	7115	2686	5199	2686
Independent reflections (Rint)	1480 (0.0348)	3567 (0.0412)	2503 (0.1074)	1393 (0.0717)	1748 (0.0449)	1393(0.0717)
Final R1, wR2[I>2σ(I)]	0.0728, 0.1853	0.0744, 0.1723	0.0637, 0.1540	0.0431, 0.1250	0.0322, 0.0819	0.0431, 0.1250

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