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The Crystal and Molecular Structure of a Dimer of 1*H*,4*H*-Naphtho[1,8]diselenepine

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The crystal and molecular structure of a dimer of 1*H*,4*H*-naphtho[1,8]diselenepine has been determined. The data used were 1350 reflexions measured on a Picker four-circle diffractometer. The crystals are monoclinic with space group *C2/c* and cell dimensions $a=22.907$, $b=5.1459$, $c=18.091$ Å and $\beta=97.97^\circ$. The structure was solved by the heavy-atom technique and refined by full-matrix least-squares to $R=0.029$. The molecule has a twofold axis of symmetry. The naphthalene rings are inclined at 46.2° to the *ac* plane while the Se atoms lie roughly in a plane parallel to the *ac* plane. Within the molecule there is a short (1.96 Å) H–H contact causing the molecule to be somewhat distorted. The Se–Se bond has the length 2.315 Å, the Se–C bonds are 1.991, the C(*sp*³)–C(*sp*²) bonds are 1.491 and the C(*sp*²)–C(*sp*²) bonds 1.396 Å. The dihedral angle at the Se–Se bond is 88.1° .

Introduction

In connexion with work on ring systems containing sulphur or selenium Biezais-Zirnis & Fredga (1971) obtained a product which they considered to be a dimer of the intended product 1*H*,4*H*-naphtho[1,8]diselenepine. The molecular weight of the compound could not be determined, neither could the absorption spectra be recorded due to low solubility. A single-crystal analysis was undertaken to clarify the situation.

Experimental

The crystals used were small pale yellow needles crystallized from boiling xylene. Preliminary cell dimensions were obtained from Weissenberg photographs which also showed the space group to be *Cc* or *C2/c*. A crystal with the dimensions $0.024 \times 0.024 \times 0.26$ mm was mounted along the needle axis (*b* axis) and used for data collection on a Picker FACS-1 automatic four-circle diffractometer. Cu *K* α radiation

(1.54051 Å, take-off angle 3.5°) from a graphite monochromator was used.

The cell dimensions were determined from 12 reflexions with 2θ between 36 and 79° by a least-squares fit. The following values (with standard deviations) were obtained:

$$\begin{aligned}a &= 22.907 \pm 0.002 \text{ \AA} \\b &= 5.1459 \pm 0.0005 \\c &= 18.091 \pm 0.002 \\\beta &= 97.97 \pm 0.01^\circ\end{aligned}$$

The data collection was performed with $\theta/2\theta$ scan with a constant scanning speed of 0.5° (2θ) per min. The scan width was 1.5° plus a dispersion correction. The background was measured for 10 seconds at each end of the scan range. Attenuators were inserted when the count rate exceeded 10000 cps. Three standard reflexions were measured after every 50 reflexions. The sum of these reflexions was used to scale the observed intensities. The maximum correction was 3.6%.

The background count was subtracted and those reflexions having an intensity exceeding 4 standard deviations (calculated as $\sigma = (C_p + C_b)^{1/2}$, where C_p is the number of counts during the peak scan and C_b is the background count during the same time) were treated as observed. Thus 1487 reflexions of the 2531 measured reflexions with $2\theta \leq 125^\circ$ were retained and L_p corrected. Absorption correction ($\mu = 95 \text{ cm}^{-1}$) was also performed. For 37 reflexions both $0k\bar{l}$ and $0k\bar{l}$ were measured and for these the average value was used. The value of $R = \sum(|F_{0k\bar{l}}| - |F_{0k\bar{l}}|) / \frac{1}{2} \sum(|F_{0k\bar{l}}| + |F_{0k\bar{l}}|)$ was 2.5% for these reflexions. Some weak reflexions forbidden by the space group extinctions were removed so that the final set of observed data comprised 1388 unique reflexions.

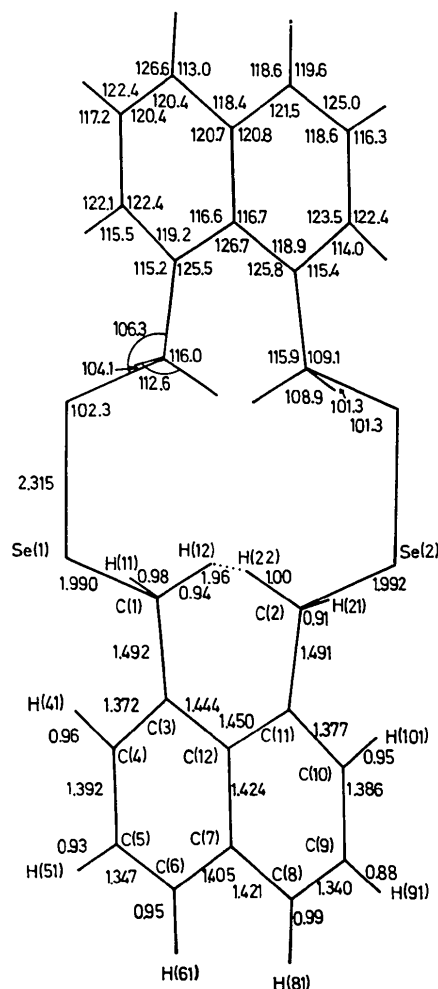


Fig. 2. Bond lengths and angles with atom numbering.

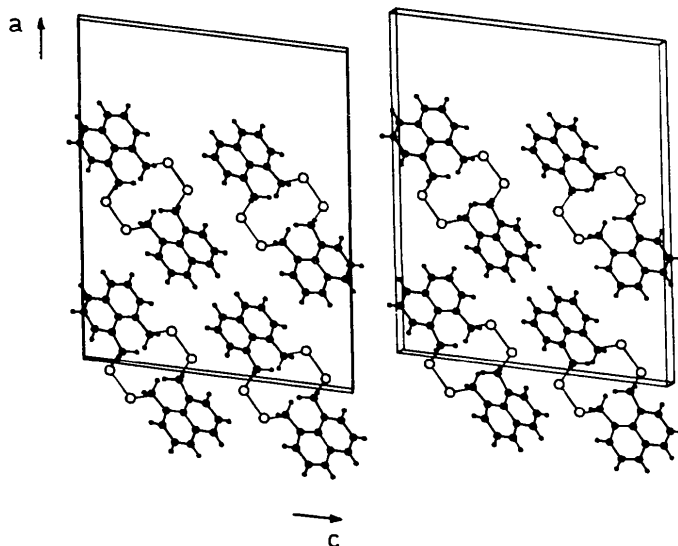


Fig. 1. Stereoscopic view of the structure seen from a direction close to the b axis.

Structure determination

Intensity statistics gave no conclusive evidence for a centre of symmetry. From the density of the crystals it was concluded that the unit cell contained 8 units of $C_{12}H_{10}Se_2$. In the space group $C2/c$ this would correspond to 8 monomeric units in general positions in the cell and in the space group Cc the structure would consist of 4 dimeric units in general positions. A Patterson function was calculated with the coefficients

Table 1. *Final atomic fractional coordinates with estimated standard deviations in parentheses*

	<i>x</i>	<i>y</i>	<i>z</i>
Se(1)	-0.10655 (4)	0.1261 (2)	0.16982 (5)
Se(2)	0.03177 (4)	0.0593 (2)	0.40199 (5)
C(1)	-0.0858 (3)	-0.1320 (16)	0.2512 (4)
C(2)	-0.0310 (4)	0.2979 (16)	0.3546 (4)
C(3)	-0.1307 (3)	-0.1200 (16)	0.3035 (4)
C(4)	-0.1772 (4)	-0.2883 (18)	0.2880 (5)
C(5)	-0.2224 (4)	-0.3096 (20)	0.3317 (6)
C(6)	-0.2205 (4)	-0.1588 (20)	0.3929 (6)
C(7)	-0.1746 (4)	0.0202 (17)	0.4124 (5)
C(8)	-0.1750 (4)	0.1711 (22)	0.4781 (5)
C(9)	-0.1336 (5)	0.3523 (21)	0.4970 (5)
C(10)	-0.0891 (4)	0.3910 (18)	0.4536 (5)
C(11)	-0.0842 (3)	0.2441 (16)	0.3911 (4)
C(12)	-0.1283 (3)	0.0477 (16)	0.3678 (4)

Table 2. *Final thermal parameters ($\times 10^4 \text{ \AA}^2$) with estimated standard deviations*

The anisotropic temperature factor is $\exp[-2\pi^2(h^2a^*2U_{11} + k^2b^*2U_{22} + l^2c^*2U_{33} + 2klb^*c^*U_{23} + 2lhc^*a^*U_{31} + 2hka^*b^*U_{12})]$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
Se(1)	396 (6)	498 (6)	365 (5)	9 (5)	23 (4)	104 (5)
Se(2)	394 (5)	446 (6)	338 (5)	44 (5)	18 (4)	-25 (5)
C(1)	414 (48)	351 (48)	439 (45)	-61 (41)	6 (36)	8 (41)
C(2)	500 (51)	325 (52)	416 (47)	4 (38)	-5 (38)	-73 (41)
C(3)	359 (47)	295 (47)	480 (47)	61 (40)	3 (36)	5 (40)
C(4)	480 (55)	471 (57)	533 (54)	25 (46)	-11 (44)	-21 (47)
C(5)	415 (57)	553 (65)	791 (70)	104 (57)	1 (50)	-148 (49)
C(6)	432 (56)	665 (68)	678 (65)	233 (60)	179 (48)	-16 (55)
C(7)	425 (51)	469 (54)	438 (49)	115 (42)	37 (38)	97 (43)
C(8)	487 (56)	725 (72)	483 (54)	153 (53)	164 (43)	191 (56)
C(9)	734 (71)	646 (70)	440 (51)	-81 (50)	98 (49)	275 (61)
C(10)	544 (55)	469 (56)	434 (47)	-52 (46)	-38 (41)	62 (47)
C(11)	398 (47)	298 (42)	379 (45)	78 (37)	2 (36)	78 (40)
C(12)	363 (47)	340 (46)	414 (44)	99 (40)	-3 (35)	92 (39)

Table 3. *Final hydrogen parameters*

Fractional coordinates and isotropic temperature factor $\exp(-B \sin^2 \theta/\lambda^2)$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
H(11)	-0.0879 (29)	-0.295 (14)	0.2229 (37)	3.2 (1.6)
H(12)	-0.0464 (26)	-0.103 (12)	0.2725 (32)	2.1 (1.4)
H(21)	-0.0167 (27)	0.459 (13)	0.3670 (34)	2.6 (1.5)
H(22)	-0.0351 (26)	0.264 (14)	0.2997 (34)	2.7 (1.4)
H(41)	-0.1747 (28)	-0.399 (13)	0.2458 (36)	3.3 (1.6)
H(51)	-0.2513 (32)	-0.434 (15)	0.3168 (39)	4.0 (1.8)
H(61)	-0.2523 (30)	-0.162 (14)	0.4213 (38)	3.9 (1.7)
H(81)	-0.2076 (31)	0.125 (14)	0.5060 (40)	4.5 (1.8)
H(91)	-0.1336 (29)	0.453 (14)	0.5363 (38)	3.4 (1.6)
H(101)	-0.0597 (32)	0.520 (15)	0.4652 (42)	4.2 (1.8)

sharpened to correspond to point atoms at rest. The map indicated a dimeric structure and the coordinates of the 4 Se atoms could be determined with Se(1) arbitrarily placed at $x=z=0$ in the space group Cc . The structure factors were calculated and gave a value of $R = \sum |F_o - F_c| / \sum |F_o| = 0.42$. One cycle of full-matrix refinement with anisotropic temperature factors gave $R = 0.36$. The corresponding electron density map revealed the positions of 7 carbon atoms.

Three following rounds of structure factor and electron density calculations gave the positions of 10, 5 and finally 2 carbon atoms.

After 4 cycles of refinement with Se atoms treated anisotropically and C atoms isotropically the value of R was 0.14. A difference electron density now showed positive density at the expected hydrogen atom positions and the hydrogen atoms were included at their calculated positions assuming a C-H distance of 1.09 Å. At this stage also a correction for anomalous dispersion by the Se atoms was introduced with $\Delta f' = 1.1$ and $\Delta f'' = -1.2$.

Some more cycles of full-matrix refinement with several adjustments of the constants in the weighting function $w = (1 + (F_o - a)^2/b^2)^{-1}$ resulted in a value of $R = 0.046$. The standard deviations were, however, high and the shifts in the parameters were up to 3.6σ . The interatomic distances and angles showed a large deviation

around their average values which were reasonable.

The molecular structure indicated that the molecule might possess a twofold axis of symmetry. The molecule was then placed with this axis coinciding with a twofold axis in the space group $C2/c$ and the refinement continued with half of the dimeric molecule as the asymmetric unit. The standard deviations now reduced to more reasonable values.

A study of the agreement between F_{obs} and F_{calc} showed the former to be about 5% too low for the 8 strongest reflexions ($F_{obs} > 300$). This could be a result of secondary extinction and a correction for this was

Table 4 (cont.)

4				4				4				4				4				4				4							
<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> _{obs} <i>F</i> _{calc}	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> _{obs} <i>F</i> _{calc}	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> _{obs} <i>F</i> _{calc}	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> _{obs} <i>F</i> _{calc}	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> _{obs} <i>F</i> _{calc}	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> _{obs} <i>F</i> _{calc}	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> _{obs} <i>F</i> _{calc}	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> _{obs} <i>F</i> _{calc}
14	2	7	229 209	15	1	-7	392 397	15	2	8	162 157	17	1	8	559 403	17	3	0	473 603	20	2	0	456 444	20	2	0	456 444	20	2	7	201 226
14	2	9	454 402	15	1	-9	420 431	15	2	10	257 247	17	1	9	191 140	17	3	1	341 307	20	2	0	456 444	20	2	0	456 444	20	2	9	494 402
14	2	10	158 405	15	1	-10	104 104	15	2	11	317 306	17	1	10	220 256	17	3	2	448 474	20	2	0	456 444	20	2	0	456 444	20	2	10	158 405
14	2	11	274 167	15	1	-11	1514 1333	15	2	12	293 238	17	1	11	102 130	17	3	3	648 609	21	3	-9	367 374	21	3	-9	367 374	21	3	-9	367 374
14	2	12	150 254	15	1	-12	358 1039	15	2	13	177 208	17	1	12	359 285	17	3	4	235 255	21	3	-7	476 480	21	3	-7	476 480	21	3	-7	476 480
14	2	13	655 862	15	1	-13	667 677	15	2	14	420 410	17	1	13	729 735	17	3	5	648 609	21	3	-5	584 622	21	3	-5	584 622	21	3	-5	584 622
14	2	14	210 263	15	1	-14	353 353	15	2	15	481 488	17	1	14	359 285	17	3	6	235 255	21	3	-7	476 480	21	3	-7	476 480	21	3	-7	476 480
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14	2	16	249 294	15	1	-16	353 353	15	2	17	453 453	17	1	16	359 285	17	3	8	331 302	21	3	-7	476 480	21	3	-7	476 480	21	3	-7	476 480
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14	2	19	412 316	15	1	-19	277 377	15	2	20	452 463	17	1	19	459 459	17	3	11	309 414	21	3	-7	476 480	21	3	-7	476 480	21	3	-7	476 480
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14	2	21	184 308	15	1	-21	261 243	15	2	22	452 454	17	1	21	459 459	17	3	13	502 502	21	3	-7	476 480	21	3	-7	476 480	21	3	-7	476 480
14	2	22	101 135	15	1	-22	472 512	15	2	23	481 450	17	1	22	459 459	17	3	14	481 481	21	3	-7	476 480	21	3	-7	476 480	21	3	-7	476 480
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15	3	0	159 517	15	2	0	482 480	15	3	0	482 480	17	2	0	482 480	17	3	0	482 480	21	4	0	482 480	21	4	0	482 480	21	4	0	482 480
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15	3	12	323 313	15	2	-12	252 252	15	3	-12	252 252	17	2	-12	252 252	17	3	-12	252 252	21	4	-12	252 252	21	4	-12	252 252	21	4	-12	252 252
15	3	-14	708 621	15	2	-14	342 334	15	3	-14	342 334	17	2	-14	342 334	17	3	-14	342 334	21	4	-14	342 334	21	4	-14	342 334	21	4	-14	342 334
15	3	-12	680 675	15	2	-12	193 197	15	3	-12	193 197	17	2	-12	193 197	17	3	-12	193 197	21	4	-12	193 197	21	4	-12	193 197	21	4	-12	193 197
15	3	-10	846 875	15	2	-10	489 510	15	3	-10	489 510	17	2	-10	489 510	17	3	-10	489 510	21	4	-10	489 510	21	4	-10	489 510	21	4	-10	489 510
15	3	-12	1375 1377	15	2	-12	485 514	15	3	-12	485 514	17	2	-12	485 514	17	3	-12	485 514	21	4	-12	485 514	21							

Results and discussion

Molecular geometry

A stereoscopic view of the structure as seen along a direction close to both the *b* axis and the twofold axis of the molecule is shown in Fig. 1. The bond lengths and angles are shown in Fig. 2 and listed in Tables 5 and 6.

Table 5. *Bond lengths*

The estimated standard deviations are for Se-Se, 0.002 Å; Se-C, 0.015 Å; C-C, 0.021 Å; C-H, 0.12 Å.

Se(1)-Se(2)	2.315 Å	C(11)-C(12)	1.450 Å
Se(1)-C(1)	1.990	C(12)-C(3)	1.444
Se(2)-C(2)	1.992	C(12)-C(7)	1.424
C(1)-C(3)	1.492	C(1)-H(11)	0.98
C(2)-C(11)	1.491	C(1)-H(12)	0.94
C(3)-C(4)	1.372	C(2)-H(21)	0.91
C(4)-C(5)	1.392	C(2)-H(22)	1.00
C(5)-C(6)	1.347	C(4)-H(41)	0.96
C(6)-C(7)	1.405	C(5)-H(51)	0.93
C(7)-C(8)	1.421	C(6)-H(61)	0.95
C(8)-C(9)	1.340	C(8)-H(81)	0.99
C(9)-C(10)	1.386	C(9)-H(91)	0.88
C(10)-C(11)	1.377	C(10)-H(101)	0.95

Average values

Se-Se	2.315 Å
Se-C	1.991
C(<i>sp</i> ³)-C(<i>sp</i> ²)	1.491
C(<i>sp</i> ²)-C(<i>sp</i> ²)	1.396
C-H	0.95

Table 6. *Bond angles*

The estimated standard deviations are for Se-Se-C, 0.4°; Se-C-C, 1.0°; C-C-C, 1.4°.

Se(2)3-Se(1)-C(1)	101.3°
Se(1)3-Se(2)-C(2)	102.3
Se(1)-C(1)-C(3)	109.1
Se(2)-C(2)-C(11)	106.3
C(1)-C(3)-C(12)	125.8
C(1)-C(3)-C(4)	115.4
C(12)-C(3)-C(4)	118.9
C(3)-C(4)-C(5)	123.5
C(4)-C(5)-C(6)	118.6
C(5)-C(6)-C(7)	121.5
C(6)-C(7)-C(12)	120.8
C(6)-C(7)-C(8)	118.4
C(12)-C(7)-C(8)	120.7
C(7)-C(8)-C(9)	120.4
C(8)-C(9)-C(10)	120.4
C(9)-C(10)-C(11)	122.4
C(10)-C(11)-C(2)	115.2
C(2)-C(11)-C(12)	125.5
C(10)-C(11)-C(12)	119.2
C(11)-C(12)-C(7)	116.6
C(7)-C(12)-C(3)	116.7
C(11)-C(12)-C(3)	126.7

Average values

Se-Se-C	101.8
Se-C-C	107.7
C-C-C	120.4

The Se atoms lie roughly in a plane parallel to the *ac* plane: two of them 0.17 Å above and the other two 0.17 Å below the plane $y=0.09269$. The two naphthalene rings are inclined at 46.2° to the *ac* plane so that the angle between the two planes is 87.6°.

The least-squares plane through the 12 carbon atoms of one half of the molecule has the equation (x , y and z are the fractional coordinates)

$$-0.73627x + 0.25147y - 0.62823z + 0.12454 = 0.$$

The direction cosines relative to the a , b , c^* directions are -0.45516 , 0.69204 and -0.56028 respectively.

The root-mean-square deviation from the plane is 0.029 Å. The maximum deviation is shown by C(8) at 0.055 Å which corresponds to 3.7σ . As C(6), however, does not show a similar deviation the effect is probably not real because in other respects the two halves of the naphthalene ring are very similar.

If the planarity of the naphthalene ring is tested with the χ^2 distribution it is found to be non-planar. Ring *A* [atoms C(3), C(4), C(5), C(6), C(7), C(12)] has the equation

$$-0.73630x + 0.25784y - 0.62561z + 0.12402 = 0$$

and ring *B* [atoms C(11), C(12), C(7), C(8), C(9), C(10)] the equation

$$-0.76333x + 0.24184y - 0.59903z + 0.11030 = 0.$$

The direction cosines are -0.45020 , 0.70179 , -0.55210 and -0.48245 , 0.68042 , -0.55161 respectively. This gives an angle of 2.2° between the two planes. The atoms of ring *A* have a root-mean-square deviation of 0.006 Å from the ring plane while those of ring *B* have 0.014 Å. The estimated standard deviation of a carbon atom is 0.015 Å. Both rings are planar according to the χ^2 distribution.

The dihedral angle around the Se-Se bond is 88.1°. This is in agreement with the value 89.5° in α, α' -diselenobisformamidinium dichloride (Chiesi Villa, Nardelli & Vidoni Tani, 1970) while some other structure determinations have given lower values (see Table 7). The dihedral angle around the bond Se(1)-C(1) is 0.7° and around Se(2)-C(2) is 7.0°. The difference is significant and shows the molecule to be irregular in that the atoms C(1) and C(3) lie at a greater distance (1.16 and 1.09 Å) below the plane $y=0.09269$ than C(2) and C(11) lie above the same plane (1.06 and 0.78 Å).

Bond lengths

Some bond lengths and angles involving Se atoms found in the literature are compiled in Table 7 and compared with the present results.

The Se-Se bond has a length of 2.315 ± 0.002 Å which is in the same range as other reported distances except for the long one (2.380 ± 0.006 Å) reported for α, α' -diselenobisformamidinium dichloride (Chiesi Villa, Nardelli & Vidoni Tani, 1970).

The Se-C bonds are 1.991 ± 0.011 Å long in good agreement with the other Se-C (aliphatic) bonds in

Table 7: 1.977 ± 0.012 Å (dimethyl selenide), 2.01 ± 0.03 Å (1,4-diselenane), 1.947 ± 0.024 and 1.980 ± 0.024 Å (1,4-diselenane. $2I_2$), 1.97 ± 0.01 Å (bisdiphenylmethyl diselenide), 1.97 ± 0.02 (*rac.*-1,2-diselenane-3,6-dicarboxylic acid), 1.96 ± 0.025 Å (tetrahydro-selenophene. I_2) and 1.962 ± 0.023 and 1.946 ± 0.016 Å (trimethylselenonium iodide). Short Se–C (aliphatic) bonds have been observed in α, α' -diselenobisformamidinium dichloride with 1.94 ± 0.01 and in *N*-acetyl-*N'*-phenylselenourea with 1.87 ± 0.03 Å. These agree with the generally shorter Se–C (aromatic) bonds (Table 7).

The $C(sp^3)$ – $C(sp^2)$ bonds are 1.491 ± 0.015 Å long and can be compared with similar bonds in bisphenylmethyl diselenide (Palmer & Palmer, 1969) where the distance is 1.52 ± 0.02 Å and in dibenzyl disulfide (Lee & Bryant, 1969) where the corresponding values are 1.49 ± 0.018 and 1.50 ± 0.016 Å. Lide (1962) gives the length 1.501 ± 0.004 Å for this type of bond.

The $C(sp^2)$ – $C(sp^2)$ bonds have an average length of 1.396 Å. There are, however, differences between the differently located bonds which are illustrated in Table 8. The average of the bonds of type 1–9 is 1.430 ± 0.010 and of type 1–2, 1.359 ± 0.010 Å. The difference is significant and the values agree well with those of Cruickshank (1962) chosen to be the best estimate of the current value based on accurate structure determinations. The bonds of types 2–3 and 9–10 have intermediate lengths and are also in reasonable agreement with Cruickshank. The mean C–H bond distance (0.95 ± 0.04 Å) is in agreement with the values usually obtained with X-ray diffraction methods.

All bond lengths mentioned are uncorrected for thermal motion. A correction for riding motion has been calculated according to Busing & Levy (1964) but the corrections were small, for the Se–Se bond 0.9σ , for Se–C maximum 0.2σ and for C–C maximum 0.8σ .

Table 7. Some bond lengths and angles involving selenium with *e.s.d.*'s in parentheses

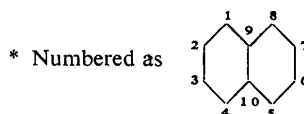
	Se–Se	Se–C	C–Se–Se	C–Se–C	Dihedral angle at Se–Se	Reference
Compounds with Se bonded to non-aromatic carbon atoms						
Present work	2.315 (2) Å	1.991 (11) Å	101.3° (4) 102.3 (4)		88.1°	
Dimethyl selenide		1.977 (12)		98° (10)		1
1,4-Diselenane		2.01 (3)		98.6 (20)		2
1,4-Diselenane . $2I_2$		1.947 (24) 1.980 (24)		100.5 (18)		3
α, α' -Diselenobisformamidinium dichloride	2.380 (6)	1.94 (1)			89.5	4
Bisdiphenylmethyl diselenide	2.285 (5)	1.97 (1)	100.1 (5)		82	5
<i>N</i> -acetyl- <i>N'</i> -phenylselenourea		1.87 (3)				6
<i>rac.</i> -1,2-Diselenane-3,6-dicarboxylic acid	2.32 (2)	1.97 (2)	96 (1)		56.0 (15)	7
Tetrahydro-selenophene . I_2		1.960 (25) 1.960 (25)		93.2 (18)		8
Trimethylselenonium iodide		1.962 (23) 1.946 (16)		97.9 (7) 99.1 (7)		9
Compounds with Se bonded to aromatic carbon atoms						
Dibenzoselenophene		1.899 (5)		86.7 (2)		10
<i>trans</i> -Selenophene		1.87 (1) 1.93 (2)		86.1 (8)		11
Diphenyl diselenide	2.29 (1)	1.93 (5)	107.5 (20) 104.6 (20)		82 (3)	12
<i>p, p'</i> -Dichlorodiphenyl diselenide	2.333 (15)	1.94 (10) 1.92 (10)			74.5	13
Di- <i>p</i> -tolyl selenide		1.920 (45) 1.930 (45)	106.2 (20)			14
Diphenylselenium dibromide		1.91 (3)		110 (10)		15
Di- <i>p</i> -tolylselenium dichloride		1.93 (3)		106.5 (10)		16
Di- <i>p</i> -tolylselenium dibromide		1.95 (3)		108 (1)		16

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- (2) Marsh & McCullough (1951).
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- (5) Palmer & Palmer (1969).
- (6) Perez-Rodriguez & López-Castro (1969).
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- (9) Hope (1966).
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- (11) Chiesi Villa, Nardelli & Palmieri (1969).
- (12) Marsh (1952).
- (13) Kruse, Marsh & McCullough (1957).
- (14) Blackmore & Abrahams (1955).
- (15) McCullough & Hamburger (1941).
- (16) McCullough & Marsh (1950).

Table 8. *Bond lengths in the naphthalene ring*

Type of bond*	Distance (observed)	Average (subgroup)	Average (group)	Distance (literature)†
1-9	‡1.444 Å } ‡1.450 } 1.405 } 1.421 }	1.447 (15) } 1.413 (15) }	1.430 (10)	1.421 (5)
	1-2			
2-3	1.392 } 1.386 }		1.389 (15)	1.415 (5)
9-10	1.424		1.424 (21)	1.418 (5)



† Cruickshank (1962).

‡ Bond adjacent to substituents.

The difference Fourier map calculated on the final parameters shows positive density in the neighbourhood of the bonds, the amount varying between 0.06 and 0.24 e.Å⁻³ near the bond centre. There is also some positive density in regions roughly corresponding to the lone pair positions at *sp*³ hybridized Se atoms.

Molecular overcrowding

The irregularity of the molecular shape mentioned above is probably caused by the close contact (1.96 Å) between the hydrogen atoms H(12) and H(22). Recently Kay, Okaya & Cox (1971) found distortions of the phenanthrene molecule resulting from a H-H contact of 2.04 Å.

The close hydrogen contact (the ordinary van der Waals distance is 2.4 Å) also causes other distortions of the molecule. Table 8 shows that the two bonds of the 1-9 type adjacent to the substituents [C(3)-C(12) and C(12)-C(11)] have an average length of 1.447 Å while the other two 1-9 bonds [C(6)-C(7) and C(7)-C(8)] give the average 1.413 Å. The estimated standard deviations of the means are 0.015 Å but as both independent bonds within each subgroup agree

Table 9. *Designation of atoms*

The numbering of the atoms is as in Fig. 2. The figures in brackets show the number of axial translations. The roman superscripts show the following symmetry operations:

i	<i>x</i>	<i>y</i>	<i>z</i>
ii	- <i>x</i>	- <i>y</i>	- <i>z</i>
iii	- <i>x</i>	<i>y</i>	$\frac{1}{2}$ - <i>z</i>
iv	<i>x</i>	- <i>y</i>	$\frac{1}{2}$ + <i>z</i>

The plus sign indicates the centering operation ($\frac{1}{2}, \frac{1}{2}, 0$).

Table 10. *The shorter non-bonded intramolecular distances (Å)*

The sums of the van der Waals radii are for Se-C 3.85 Å and for C-C 3.70 Å.

Outside the naphthalene ring		Within the naphthalene ring				
Se(1)	C(2 ⁱ)	3.65	C(3)	C(5 ⁱ)	2.43	
	C(2 ⁱⁱⁱ)	3.36		C(6 ⁱ)	2.79	
	C(3 ⁱ)	2.85		C(7 ⁱ)	2.44	
	C(4 ⁱ)	3.56		C(8 ⁱ)	3.76	
Se(2)	C(12 ⁱ)	3.71		C(10 ⁱ)	3.81	
	C(1 ⁱ)	3.69		C(11 ⁱ)	2.59	
	C(1 ⁱⁱⁱ)	3.34	C(4)	C(6 ⁱ)	2.35	
	C(10 ⁱ)	3.49		C(7 ⁱ)	2.75	
C(1)	C(11 ⁱ)	2.80		C(11 ⁱ)	3.80	
	C(12 ⁱ)	3.63		C(12 ⁱ)	2.43	
	C(1 ⁱⁱⁱ)	3.94	C(5)	C(7 ⁱ)	2.40	
	C(2 ⁱ)	3.05		C(8 ⁱ)	3.68	
C(2)	C(4 ⁱ)	2.42		C(12 ⁱ)	2.84	
	C(5 ⁱ)	3.74	C(6)	C(8 ⁱ)	2.43	
	C(7 ⁱ)	3.86		C(9 ⁱ)	3.66	
	C(11 ⁱ)	3.18		C(11 ⁱ)	3.75	
C(7)	C(12 ⁱ)	2.61		C(12 ⁱ)	2.46	
	C(3 ⁱ)	3.18	C(7)	C(9 ⁱ)	2.40	
	C(7 ⁱ)	3.86		C(10 ⁱ)	2.76	
	C(9 ⁱ)	3.73		C(11 ⁱ)	2.45	
C(8)	C(10 ⁱ)	2.42		C(10 ⁱ)	2.37	
	C(12 ⁱ)	2.61		C(11 ⁱ)	2.80	
				C(12 ⁱ)	2.47	
				C(9)	C(11 ⁱ)	2.42
				C(12 ⁱ)	2.83	
				C(10)	C(12 ⁱ)	2.44

very well there seems to be a significant lengthening of the two bonds on the substituent side of the molecule. The same argument applies to the 1-2 bonds with values 1.375 and 1.344 Å. Consideration of the angles at C(3) and C(11) shows that there are significant differences from 120° (the estimated standard

deviation in C–C–C angles is 1.4°. The bonds C(3)–C(1) and C(11)–C(3) are thus diverging so that the distance C(1)–C(2) is 3.05 Å while C(3)–C(11) is 2.59 Å.

Other distortions occur at C(12) where the angle C(3)–C(12)–C(11) is 126.7° and at C(4) and C(10) where the angles within the ring are 123.5° and 122.4°. It thus seems as if the strain at C(3) and C(11) resulting from forcing C(1) and C(2) apart is partly relieved by turning the bonds C(3)–C(4) and C(3)–C(12) around C(3) in the same direction as C(3)–C(1). In the same way C(11)–C(10) and C(11)–C(12) are turned around C(11) in the same direction as C(11)–C(2). A deformation of the same type but in opposite direction has recently been described by Prout, Cameron, Dunn, Hodder & Viterbo (1971) for 1,8-dinitrosonaphthalene.

With the designations of the atoms according to Table 9 the shorter non-bonded distances within the molecule are listed in Table 10. The range of Se–C contacts goes down to 2.80 Å for Se(2)–C(11) and 2.85 Å for Se(1)–C(3). The shortest C–C contacts (except for those in the naphthalene ring) are C(1)–C(4) and C(2)–C(10) both 2.42 Å.

Molecular packing

The molecular packing is evident from Fig. 1. In Table 11 the shorter intermolecular contacts are listed with designation of the atoms according to Table 9. At Se(1) there are no carbon contacts shorter than the

Table 11. *The shorter intermolecular distances*

The sums of the van der Waals radii are for Se–C, 3.85 Å and for C–C, 3.70 Å.

Se(1)	C(5 ⁱⁱⁱ⁺) [T00]	3.93 Å
	C(8 ^{iv}) [00T]	3.91
Se(2)	C(9 ^{iv}) [00T]	3.96
	C(8 ⁱⁱ) [001]	3.86
	C(9 ⁱⁱ) [001]	3.48
C(1)	C(10 ⁱⁱ) [001]	3.60
	C(10 ⁱⁱ) [011]	3.95
	C(2 ⁱ) [0T0]	3.61
C(2)	C(3 ⁱ) [010]	3.80
C(3)	C(10 ⁱ) [0T0]	3.73
	C(11 ⁱ) [0T0]	3.73
C(4)	C(5 ⁱⁱⁱ⁺) [T00]	3.83
	C(5 ⁱⁱⁱ⁺) [T10]	3.97
	C(10 ⁱ) [0T0]	3.76
C(5)	C(11 ⁱ) [0T0]	3.57
	C(12 ⁱ) [0T0]	3.82
	C(5 ⁱⁱⁱ⁺) [T00]	3.99
	C(5 ⁱⁱⁱ⁺) [T10]	3.99
	C(7 ⁱ) [0T0]	3.84
	C(8 ⁱ) [0T0]	3.81
	C(9 ⁱ) [0T0]	3.80
	C(10 ⁱ) [0T0]	3.83
	C(11 ⁱ) [0T0]	3.94
	C(12 ⁱ) [0T0]	3.95
C(6)	C(8 ⁱ) [0T0]	3.86
	C(9 ⁱ) [0T0]	3.58
	C(10 ⁱ) [0T0]	3.84
C(7)	C(9 ⁱ) [0T0]	3.83
	C(10 ⁱ) [0T0]	3.80
C(8)	C(8 ⁱⁱⁱ⁺) [T01]	3.72
	C(12 ⁱ) [010]	3.78

sum of the van der Waals radii (3.85 Å). At Se(2) there are two short contacts with carbon atoms namely 3.48 and 3.60 Å. This again illustrates that the two halves of the monomeric unit are not equivalent either in molecular geometry or in packing. The carbon–carbon intermolecular contacts range down to 3.57 Å which is only slightly below the sum of the van der Waals radii 3.70 Å. Looking at the contacts between the hydrogens one finds the shortest distance 2.31 Å between H(81) and H(81ⁱⁱⁱ⁺) [T01] (see Table 9) while all other contacts are longer than 2.8 Å. Here the refined hydrogen positions are used for calculating the distances and the average C–H distance is only 0.95 Å. Assuming a normal distance of 1.09 Å the shortest contact would be 2.18 Å and all the others above 2.6 Å. No significant deformations can be seen in the parts of the molecule which are engaged in the shorter intermolecular contacts.

Table 12. *Magnitude and direction cosines of the principal axes of the vibration ellipsoids*

	Direction cosines			
	<i>B</i>	<i>a</i>	<i>b</i>	<i>c</i> *
Se(1)	3.02	0.305	0.535	0.788
	4.46	−0.317	0.837	−0.445
	2.53	−0.898	−0.114	0.425
Se(2)	3.06	−0.844	−0.404	0.353
	3.80	−0.505	0.821	−0.268
	2.51	0.182	0.404	0.896
C(1)	3.08	−0.862	−0.470	0.188
	4.03	0.374	−0.342	0.862
	2.52	−0.341	0.814	0.471
C(2)	4.49	0.827	−0.425	0.369
	3.11	−0.276	0.266	0.924
	2.35	−0.490	−0.865	0.103
C(3)	2.77	−0.940	−0.257	−0.224
	4.14	−0.288	0.244	0.926
	2.16	−0.183	0.935	−0.303
C(4)	3.65	−0.051	−0.519	0.853
	4.85	−0.967	0.240	0.088
	3.40	0.250	0.820	0.514
C(5)	2.52	0.832	−0.239	−0.501
	7.04	0.553	0.428	0.715
	4.51	−0.044	0.872	−0.488
C(6)	4.07	−0.700	0.158	0.696
	7.15	0.584	0.687	0.432
	2.55	−0.411	0.709	−0.573
C(7)	3.60	−0.719	0.399	0.570
	4.63	−0.075	0.770	−0.633
	2.34	0.691	0.498	0.524
C(8)	3.25	−0.354	0.469	0.809
	7.02	0.531	0.813	−0.239
	2.89	−0.770	0.345	−0.537
C(9)	2.60	−0.408	0.737	−0.539
	7.74	0.611	0.659	0.438
	3.99	0.678	−0.151	−0.719
C(10)	3.00	0.500	0.734	0.460
	5.23	0.223	0.404	−0.887
	3.43	0.837	−0.546	−0.039
C(11)	3.35	−0.371	0.808	0.458
	3.56	−0.608	0.162	−0.778
	1.69	−0.702	−0.567	0.431
C(12)	3.45	−0.817	0.024	0.576
	3.81	−0.478	0.531	−0.700
	1.67	0.323	0.847	0.422

Thermal motion

In the thermal parameters given in Table 2 one can see a tendency to anisotropic vibrations. The magnitudes and direction cosines of the principal axes of the vibration ellipsoids after transformation to the orthogonal coordinate system *a*, *b*, *c** are shown in Table 12. For the Se atoms the direction of maximum vibration is directed roughly along the *b* axis while the carbon atoms of the naphthalene ring have their maximum vibration roughly perpendicular to the ring plane. The anisotropy is most marked for atoms C(5), C(6), C(8) and C(9).

The hydrogen atoms have *B* values which on the average are 0.6 units lower than the equivalent isotropic temperature factor of the parent atoms (3.4 and 4.0). The difference is, however, hardly significant.

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