Crystal Structure of a [1,2,5]Oxaselenazolo[2,3-*b*][1,2,5]oxaselenazole-7-*Se*^{IV}: A Molecule with ' Short ' Intramolecular Se $\cdots$ O Distances, or ' Long ' Se–O Bonds

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The crystal structure of the title compound (IV) has been determined from *X*-ray diffraction by Patterson and Fourier methods. Crystals are monoclinic with a = 5·837(2), b = 20·337(11), c = 7·971(2) Å, and β = 97° 55(2′), space group $P2_1/n$, with Z = 4. The structure, including hydrogen atoms, has been refined to R 0·056 for 1154 non-zero reflections. The non-hydrogen atoms in the molecule are almost coplanar except for the CMe_2 group in the six-membered ring. The selenium atom interacts with the oxygen atoms of the two nitroso-groups in the molecule to give Se–O 2·017(9) and 2·030(9) Å; O–Se–O is 199·2(4)°. The lengths of the Se–O, C–Se, C–C, C–N, and N–O bonds all support a bicyclic structure for the O–Se–O–N–C–C–C–N moiety involving three-co-ordinate selenium. In the crystal, the molecules of (IV) are arranged as centrosymmetric dimers with two Se $\cdots$ O intermolecular distances of 3·20 Å.

STUDIES [1-4] of molecules (I) where oxygen is substituted for a terminal sulphur in the thiathiophthen structure have shown that the closest S $\cdots$ O distance occurs when the oxygen atom belongs to a nitroso-group. In cases where R^3 contains a competing oxygen atom [(Id) and (Ie)], there is a strong preference for the nitroso-group.[2] These results suggest that a molecule of type

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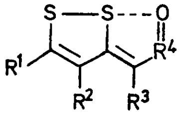
[1] R. J. S. Beer and R. J. Gait, *Chem. Comm.*, 1970, **328**; R. J. S. Beer, D. Cartwright, R. J. Gait, and D. Harris, *J. Chem. Soc.* (*C*), 1971, **963**.

[2] P. L. Johnson and I. C. Paul, *J. Amer. Chem. Soc.*, 1969, **91**, 781; P. L. Johnson, K. I. G. Reid, and I. C. Paul, *J. Chem. Soc.* (*B*), 1971, 946.

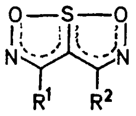
[3] K. I. G. Reid and I. C. Paul, *Chem. Comm.*, 1970, **329**; *J. Chem. Soc.* (*B*), 1971, **952**.

[4] A. Hordvik, E. Sletten, and J. Sletten, *Acta Chem. Scand.*, 1969, **23**, **1377**.

(II), where two external nitroso-groups form a bicyclic structure with a four-covalent sulphur atom, might be stable. No compound of this type with nitroso-groups has yet been synthesized but a selenium analogue exists.[5] The structure of the fusion product of captan with resorcinol [6] has recently been shown to have structure (III),[7] with S–O lengths of 1·878(2) and 1·879(2) Å.

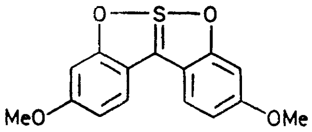


(I)

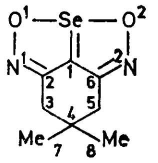


(II)

a; $R^1 = R^2 = R^3 = H$, $R^4 = N$(nitroso)
b; $R^1 = R^2 = R^3 = H$, $R^4 = NO$(nitro)
c; $R^1 = R^2 = R^3 = H$, $R^4 = CR^5$(carbonyl); $R^5 = Me$ or H
d; $R^1 = Ph$, $R^2 = H$, $R^3 = C(\colon O)Ph$, $R^4 = N$
e; $R^1 = Ph$, $R^2 = H$, $R^3 = -NO_2$, $R^4 = N$



(III)



(IV)

We now report the results of an X-ray structural study of the [1,2,5]oxaselenazolo[2,3-*b*][1,2,5]oxaselenazole-7-$\mathit{Se}^{\mathrm{IV}}$ molecule (IV), a preliminary report of which has appeared.[8]

EXPERIMENTAL

Crystals of (IV) were red needles elongated along the *a*-axis. After a few weeks the crystal used for data collection was observed to turn yellow.

Crystal Data.—$C_8H_{10}O_2N_2Se$, $M = 245·0$. Monoclinic, $a = 5·837(2)$, $b = 20·337(11)$, $c = 7·971(2)$ Å, $\beta = 97° 55'(2')$, $U = 937·1 \times 10^{-24}$ cm^{-3}, D_m (by flotation) $= 1·76$, $Z = 4$, $D_c = 1·74$, $F(000) = 488$. Cu-K_α radiation, $\lambda = 1·54178$ Å, μ(Cu-K_α) $= 58·9$ cm^{-1}. Space group $P2_1/n$ from systematic absences: $h0l$ when $h + l = 2n + 1$; $0k0$ when $k = 2n + 1$.

Unit-cell dimensions were determined from a least-squares fit to the angular settings for 10 reflections carefully centred on a diffractometer. Cell dimensions in the $P2_1/c$ setting would be $a = 9·207$, $b = 20·337$, $c = 7·971$ Å, and $\beta = 141° 06'$.

Data were collected by a computer-controlled Picker FACS 1 diffractometer in the 2θ sphere 0—130°. The rate of scan in 2θ was 1° min^{-1} with a basewidth for the scan of 2°. A 10 s stationary background count was made at each limit of the scan. A dispersion factor, to account for $\alpha_1 - \alpha_2$ splitting, was used and attenuators were inserted whenever the counting rate exceeded 10,000 counts s^{-1}. Of 1752 unique reflections scanned, 1154 were considered above background based on the criteria that the net count exceed 0·05 of the total background and/or 50 counts, whichever was greater. The data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by Patterson–heavy-atom methods. The co-ordinates of the selenium atom were determined from a Patterson map. A structure-factor calculation phased on the position of the selenium atom yielded R 0·38. The positions of all the other non-hydrogen atoms were found from a Fourier map based on this calculation. The positional and isotropic thermal parameters of all the non-hydrogen atoms were then refined by full-matrix least-squares methods to R 0·11. All non-zero reflections were given unit-weight and the quantity minimized was $\Sigma w(|F_o| - |F_c|)^2$. Subsequent refinements using anisotropic thermal parameters reduced R to 0·063. An electron-density difference-Fourier map calculated at this stage indicated the positions of four hydrogen atoms. A second difference-Fourier map based on all the heavy atoms and these four hydrogen atoms revealed the positions of the other six hydrogen atoms. Refinements, in which anisotropic temperature factors were varied for the thirteen non-hydrogen atoms and isotropic temperature factors varied for the hydrogen atoms, resulted in an unacceptable C–H bond length and H–C–H bond angle in the case of H(5B). The parameters of this hydrogen atom were then held constant at the positions found in the difference map in the final refinements. Further full-matrix least-squares refinement gave a final R of 0·056 on the 1154 non-zero reflections; observed and calculated structure factors are listed in Supplementary Publication No. SUP 20462 (3 pp., 1 microfiche).* There were no features in the difference-Fourier map calculated to locate the hydrogen atoms which would indicate the presence of any significant amounts of electron density not included in the model. The calculated values for the unobserved reflections did not indicate any anomalies. The scattering curves for selenium, carbon, nitrogen, and oxygen were taken from ref. 9, and that for

TABLE 1

Final atomic co-ordinates in fractions of cell edge, with estimated standard deviations in parentheses

	x	y	z
Se	0·7263(2)	0·4259(1)	0·5618(2)
O(1)	0·5421(16)	0·3628(4)	0·6856(9)
O(2)	0·8312(16)	0·4800(4)	0·3723(11)
N(1)	0·3616(20)	0·3364(6)	0·5872(12)
N(2)	0·6978(20)	0·4713(5)	0·2223(13)
C(1)	0·5055(20)	0·4034(5)	0·3906(12)
C(2)	0·5198(20)	0·4313(6)	0·2319(13)
C(3)	0·3399(23)	0·4140(6)	0·0870(14)
C(4)	0·2359(19)	0·3455(6)	0·1116(13)
C(5)	0·1519(22)	0·3376(6)	0·2878(14)
C(6)	0·3407(19)	0·3588(5)	0·4273(13)
C(7)	0·4195(24)	0·2922(6)	0·0903(17)
C(8)	0·0271(26)	0·3353(8)	−0·0261(18)
H(3A) *	0·240(24)	0·447(6)	0·080(18)
H(3B)	0·407(17)	0·417(5)	−0·039(13)
H(5A)	−0·040(26)	0·362(7)	0·285(18)
H(5B) †	0·055	0·293	0·269
H(7A)	0·361(21)	0·248(6)	0·100(15)
H(7B)	0·485(22)	0·298(6)	−0·009(17)
H(7C)	0·594(21)	0·302(6)	0·181(15)
H(8A)	0·062(22)	0·341(6)	−0·142(17)
H(8B)	−0·051(19)	0·283(6)	−0·042(14)
H(8C)	−0·094(20)	0·354(5)	−0·001(13)

* Hydrogen atoms have the same number as the carbon atoms to which they are attached. † The co-ordinates of this atom were not varied during least-squares refinement.

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* For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20 (items less than 10 pp. are sent as full size copies).

[5] F. E. King and D. G. I. Felton, *J. Chem. Soc.*, 1949, 274.

[6] I. Pomerantz, L. Miller, E. Lustig, D. Mastbrook, E. Hansen, R. Barron, N. Oates, and J.-Y. Chen, *Tetrahedron Letters*, 1969, 5307.

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[8] R. J. S. Beer, J. R. Hatton, E. C. Llaguno, and I. C. Paul, *Chem. Comm.*, 1971, 594.

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hydrogen from ref. 10. The scattering curve for selenium was corrected for the effects of anomalous dispersion.[11] Final atomic co-ordinates are listed in Table 1, while Table 2 contains final thermal parameters.

TABLE 2

Final thermal parameters with estimated standard deviations in parentheses *

	β_{11} ($\times 10^3$)	β_{22} ($\times 10^4$)	β_{33} ($\times 10^3$)	β_{12} ($\times 10^4$)	β_{13} ($\times 10^4$)	β_{23} ($\times 10^4$)
Se	34(0)	21(0)	11(0)	−1(1)	−4(2)	−7(1)
O(1)	44(4)	35(3)	7(1)	−14(8)	17(17)	6(5)
O(2)	44(4)	23(2)	16(2)	−47(8)	−15(20)	2(5)
N(1)	39(4)	31(3)	10(2)	−8(10)	74(22)	8(6)
N(2)	43(5)	20(3)	16(2)	−26(10)	1(24)	19(6)
C(1)	27(4)	15(3)	8(2)	5(8)	16(20)	0(5)
C(2)	31(4)	14(3)	10(2)	1(9)	24(21)	9(6)
C(3)	35(5)	16(3)	11(2)	−7(10)	−4(23)	15(6)
C(4)	24(4)	19(3)	9(2)	0(9)	7(21)	2(6)
C(5)	26(4)	28(4)	10(2)	−15(10)	32(22)	2(7)
C(6)	27(4)	19(3)	10(2)	3(9)	50(22)	3(6)
C(7)	31(5)	19(4)	16(2)	14(10)	81(28)	−3(7)
C(8	27(5)	40(5)	12(2)	−10(13)	−16(28)	−10(9)

	B		B
H(3A)	5(3)	H(7B)	3(3)
H(3B)	3(2)	H(7C)	5(3)
H(5A)	7(4)	H(8A)	4(3)
H(5B) †	3	H(8B)	9(3)
H(7A)	4(3)	H(8C)	6(2)

* Anisotropic thermal parameters are expressed as:

$$\exp-[\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl]$$

and the isotropic thermal parameter is in the form:

$\exp[-B \sin^2 \theta/\lambda^2]$. † The isotropic temperature factor for this hydrogen was not varied throughout the refinement.

RESULTS AND DISCUSSION

Figure 1 is a stereoscopic view of the molecular structure of (IV) showing the bond lengths between the non-hydrogen atoms. Bond lengths and angles with estimated standard deviations are given in Table 3. The results of some best planes in the molecule are shown in Table 4. The molecule is almost planar (max. devn. 0·036 Å) with the exception of the carbon atom [C(4)] containing the two methyl groups and the two methyl carbon atoms [C(7) and C(8)].

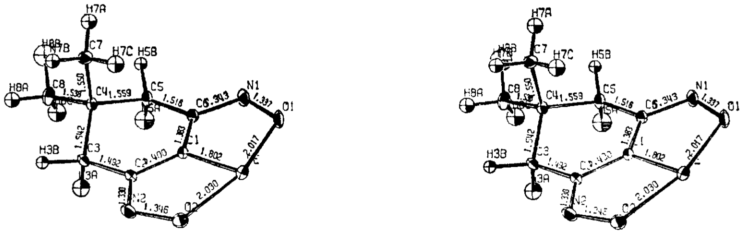

FIGURE 1 Stereoscopic drawing of a single molecule

TABLE 3

Molecular geometry, with estimated standard deviations in parentheses [a]

(*a*) Bond lengths (Å)

Se–O(1)	2·017(9)	C(4)–C(5)	1·559(15)
Se–O(2)	2·030(9)	C(5)–C(6)	1·516(16)
O(1)–N(1)	1·337(14)	C(1)–C(6)	1·383(16)
O(2)–N(2)	1·346(14)	C(4)–C(7)	1·550(18)
Se–C(1)	1·802(11)	C(4)–C(8)	1·538(18)
C(1)–C(2)	1·400(14)	C(2)–N(2)	1·330(16)
C(2)–C(3)	1·492(16)	C(6)–N(1)	1·343(14)
C(3)–C(4)	1·542(16)		

(*b*) Bond angles (deg.) [b,c]

O(1)–Se–O(2)	199·2(4)	C(3)–C(2)–N(2)	124·9(7)
O(1)–Se–C(1)	80·5(4)	C(2)–C(3)–C(4)	111·3(9)
O(2)–Se–C(1)	80·3(4)	C(3)–C(4)–C(5)	112·7(7)
Se–O(1)–N(1)	113·5(5)	C(3)–C(4)–C(7)	109·1(1·0)
Se–O(2)–N(2)	113·2(5)	C(3)–C(4)–C(8)	108·6(1·0)
O(1)–N(1)–C(6)	112·5(6)	C(5)–C(4)–C(7)	108·5(1·0)
O(2)–N(2)–C(2)	112·9(6)	C(5)–C(4)–C(8)	108·2(9)
Se–C(1)–C(2)	117·1(8)	C(7)–C(4)–C(8)	108·6(1·0)
Se–C(1)–C(6)	116·9(9)	C(4)–C(5)–C(6)	110·1(1·1)
C(2)–C(1)–C(6)	125·9(6)	C(1)–C(6)–C(5)	119·2(6)
C(1)–C(2)–C(3)	118·7(7)	C(1)–C(6)–N(1)	116·6(1·1)
C(1)–C(2)–N(2)	116·4(1·1)	C(5)–C(6)–N(1)	124·2(9)

[a] C–H range 0·85—1·23 Å, with estimated standard deviations 0·10—0·14 Å. [b] C–C–H range 100—131°, with estimated standard deviations 5—9°. [c] H–C–H range 83—117°, with estimated standard deviations 7—11°.

The lengths of the Se–O, C–Se, C–C, C–N, and N–O bonds are indicative of a structure of type (IV), although structures (V)—(VIII) probably contribute to the overall resonance hybrid. While the Se–O lengths of 2·017(9) and 2·030(9) Å are almost equal to the S · · · O(nitroso) distance of 2·034(5) Å found in (Id),[2] they probably correspond to a stronger interaction. Pauling [12] gives values of 1·04 and 1·17 Å for the covalent radii of sulphur and selenium. A difference of 0·115 Å was also found between the S–Se and S–S distances corresponding to the 'short' bond in the unsymmetrical thiathiophthens (IXa) [13] and (IXb).[14] Thus the Se–O distances in (IV) are more nearly comparable to S–O distances of 1·89—1·92 Å, values which agree well with the distances found in (III),[7] and also with the S–O distances [1·889(4) and 1·916(4) Å] found in a diaryldialkoxysulphurane.[15] The Se–O(H) distances in two benzeneseleninic acids are 1·765(15) and 1·79(5) Å,[16,17] while in SeO_2, the longer Se–O distances were 1·78(3) Å; [18] a length of 1·82(2) Å

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[10] 'International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962, pp. 201—209.

[11] Ref. 10, pp. 213—216.

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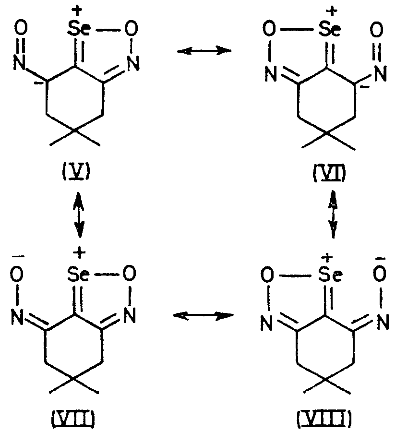
TABLE 4

Distances (Å) of atoms from various best planes in the molecule of (IV) [a]

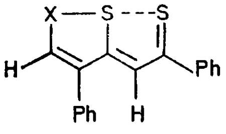
Plane	(*A*)	(*B*)	(*C*)
Se	*−0·001*	0·009	*−0·001*
O(1)	*0·013*	0·016	*0·012*
O(2)	*0·003*	0·020	*0·005*
N(1)	*0·018*	0·024	*0·021*
N(2)	*0·030*	0·051	*0·036*
C(1)	*−0·020*	*−0·007*	*−0·015*
C(2)	*−0·011*	*0·009*	*−0·003*
C(6)	*−0·010*	*0·002*	*−0·003*
C(3)	−0·029	*−0·006*	*−0·017*
C(4)	0·597	0·618	0·610
C(5)	−0·013	*0·002*	*−0·002*
C(7)			2·139
C(8)			0·366
χ^2 [b]	17·9	1·20	21·2
P [c]	<0·01	0·50	~0·01

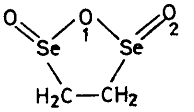
[a] Distances of atoms included in best planes calculation are italicised. [b] Atoms were weighted as $\frac{1}{\sigma^2}$ where $\sigma = 1·5 \times \sigma$ (from least-squares results). [c] The probability that the deviations from the best planes describe a normal distribution.

was found for the longer (and presumably mainly single) Se–O bond in *trans*-ethanediseleninic anhydride (X).[19]



The C–Se length [1·802(11) Å] is shorter than any C–Se length thus far reported. The lengths of Se–C(Ph) bonds lie in the range 1·91—1·94 Å,[20] while in selenophene derivatives the lengths are in the range 1·85—1·87 Å.[21] In diselenouracil, where by analogy with compounds containing N–C(:S)·N and N·C(:S)·C:C groups,[22] some considerable degree of double-bond character could be expected in the C–Se bond, the two lengths were 1·89(5) and 1·99(5) Å.[23] In 2-selenouracil, the C–Se length was 1·85 Å.[24] If one takes Pauling's values [12] for the covalent radii of singly and doubly bound carbon and selenium, the lengths of C–Se and C=Se bonds should be 1·94 and 1·74 Å, respectively. It would therefore appear that the length of 1·802(11) Å found in (IV) implies considerable double-bond character. The only other comparably short C–Se bond is in (IXa) [1·82(1) Å].[13]


(IXa) X = Se
(IXb) X = S


(X)

The C(1)–C(2) and C(1)–C(6) lengths are close to the value normally found for aromatic C–C bonds. The C–N lengths of 1·343(14) and 1·330(16) Å are intermediate between the values found for pure C=N double bonds in oxime *O*-picryl ethers [1·291(6) Å] [25] or in benzylideneaniline derivatives [1·237(3)—1·281(12) Å],[26] and those obtained for N–C(aromatic) single bonds [1·443(5)—1·448(10) Å] in phenylazotribenzoylmethane and derivatives thereof.[27] The N–O lengths [1·337(14) and 1·346(14) Å] in the nitroso-groups agree well with the value [1·312(6) Å] found in (Id) [2] and are much longer than those [1·259(4) and 1·269(4) Å] found in the *cis*-dimer of nitrosobenzene [28] or in the *trans*-dimer of *o*-nitrosobenzoic acid [1·270(4) Å].[29] The foregoing evidence clearly favours a structure with bond order between 1 and 2 for the C–Se, C–N, C–C, and N–O bonds, and between 0 and 1 for the Se–O bonds. Similar conclusions could be drawn from a consideration of the dimensions obtained for the molecule (III).[7] There are several examples reported [30,31] where selenium forms bonds to halogens and also, in the case of the addition compound of selenium oxychloride and pyridine,[31] to nitrogen with lengths considerably greater than the accepted single-bond values.

The C–N–O angles in (IV) [112·5(6) and 112·9(6)°] are much smaller than those in nitrosobenzene [120·1(3) and 120·8(3)°],[28] *o*-nitrosobenzoic acid [120·0(2)°],[29] or (Ie) [C–N–O(nitro) 117(1)°],[3] but are similar to the value [112·7(3)°] found for the C–N–O(nitroso) angle in (Id).[2] The C(2)–C(1)–C(6) angle is 125·9(6)°; a large C–C(S)–C angle [132·3(2)°] was also found in (III).[7]

The bond lengths and angles involving selenium in (IV) are quite different from those found in benzeneseleninic acid,[16] where the selenium atom has a pyramidal configuration, with Se–C 1·90, Se–O 1·71—1·77 Å, and O–Se–O 103·5°. In (IV), the O–Se–O

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angle is 199·2(4)°, and the deviation of Se from the plane defined by its three bonded neighbours is only 0·015 Å.

A stereoscopic drawing of the molecular packing, as viewed along the *a* axis is shown in Figure 2. Some important intermolecular contacts are given in Table 5:

TABLE 5

Intermolecular distances <3·8 Å for contacts not involving Se, <4·0 Å for those involving Se

O(1) ··· C(3^{I})	3·71	N(1) ··· C(8^{V})	3·77
O(1) ··· C(7^{I})	3·69	N(2) ··· C(3^{VI})	3·38
N(1) ··· C(8^{I})	3·87	Se ··· O(2^{VII})	3·87
Se ··· C(5^{II})	3·96	Se ··· N(2^{VII})	3·83
O(2) ··· C(5^{II})	3·56	Se ··· C(1^{VII})	3·76
N(2) ··· C(5^{II})	3·78	Se ··· C(2^{VII})	3·72
Se ··· C(8^{III})	3·96	O(1) ··· N(2^{VII})	3·76
O(1) ··· C(8^{III})	3·44	O(2) ··· C(1^{VII})	3·75
N(1) ··· C(7^{IV})	3·68	Se ··· O(2^{VIII})	3·20

Superscripts in Roman numerals refer to an equivalent molecule relative to the molecule at x, y, z:

I $x, y, 1 + z$
II $1 + x, y, z$
III $1 + x, y, 1 + z$
IV $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$
V $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$
VI $1 - x, 1 - y, -z$
VII $1 - x, 1 - y, 1 - z$
VIII $2 - x, 1 - y, 1 - z$

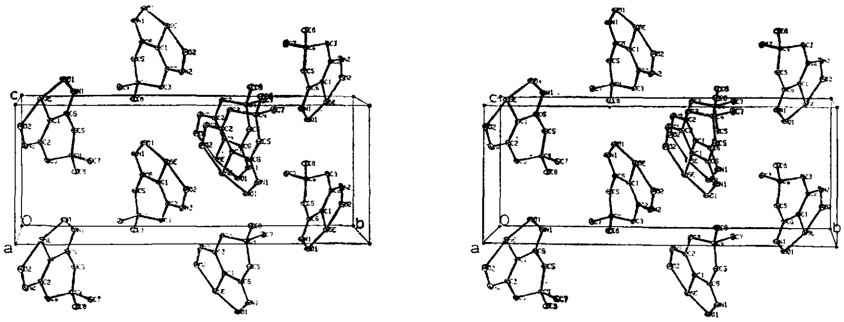

FIGURE 2 Stereoscopic drawing of the packing of molecules of (IV) in the unit cell. The Se ··· O intermolecular interaction between centrosymmetrically related molecules can be seen for two molecules near the centre of the drawing

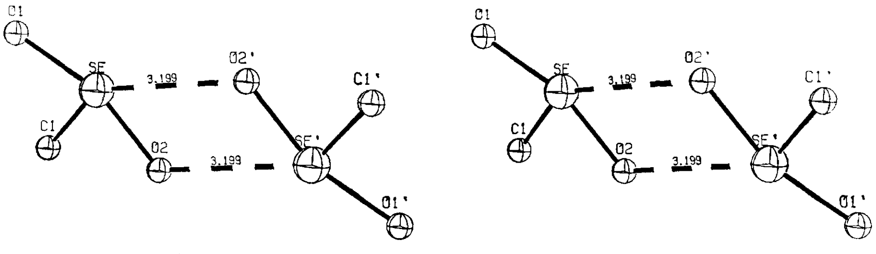

FIGURE 3 Stereoscopic drawing of the environment of the selenium atom in (IV) and also showing the Se ··· O intermolecular contact

the only one which is less than the sum (3·40 Å) of the van der Waals radii[32] for the two atoms involved is Se ··· O(2^{VIII}) 3·20 Å. This oxygen atom lies almost in the plane of the selenium atom and its three bonded neighbours. The distance of the oxygen atom from the plane defined by the intramolecularly bonded C(1), O(1), and O(2) is 0·094 Å, the deviation being on the same side of the plane as that of the selenium atom. The C–Se ···-O(2^{VIII}) angle is 138·8°. A stereoscopic drawing of the environment of the selenium atom in (IV) is shown in Figure 3. Two of these Se ··· O interactions hold the molecules of (IV) as centrosymmetrically related dimers (Figure 2). The crystal packing consists of stacks of such dimers running along the *a* direction, with the planes [*i.e.* plane (*C*) in Table 4] of the molecules making an angle of 37° 32′ with the *a* axis.

A somewhat similar Se ··· O intermolecular interaction was noted in crystals of benzeneseleninic acid,[16] in which the molecules were held as dimers by O–H ··· O hydrogen bonding. In addition, however, there was an Se ··· O intermolecular contact of 3·16 Å. The environment of the selenium atom in benzeneseleninic acid is shown in Figure 4. In this case, the Se ··· O intermolecular interaction can be seen to be an almost linear extension of the C–Se bond. Another molecule which shows short Se ··· O contacts, is *trans*-ethanediseleninic anhydride (X).[19] The arrangement of atoms around selenium in (X) is shown in Figure 5. There are two Se ··· O distances of 2·70 and 3·11 Å, respectively. In (X), the two oxygen atoms involved in these contacts are disposed such that the arrangement of atoms around selenium constitutes a distorted trigonal bipyramid.

The molecule of (IV) has effective C_s symmetry through a plane perpendicular to that containing most of the atoms in the molecule and passing through C(1) and Se. This symmetry was implied by the n.m.r. spectrum[8] which showed two sharp signals, one for the protons of the two methyl groups and one for those of the two methylene groups. The carbon atom [C(4)] bearing the *gem*-dimethyl group lies 0·610 Å out of the plane through the other five atoms [C(1)—(3), C(5), C(6)] in

[32] Ref. 12, pp. 257—264.

Figure 4 Stereoscopic drawing of the environment of the selenium atom in benzeneseleninic acid (co-ordinates from ref. 16). The distance O(1) · · · O(2′) (2·518 Å) corresponds to a hydrogen bond, while Se · · · O(1′) is 3·164 Å

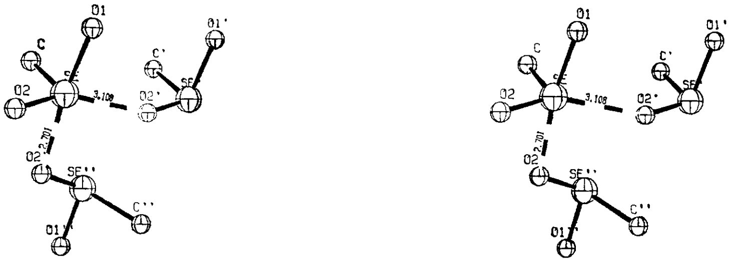

Figure 5 Stereoscopic drawing of the environment of the selenium atom in *trans*-ethanediseleninic anhydride (X) (co-ordinates from ref. 19). The approximately trigonal bipyramidal arrangement of five atoms around the nearest selenium atom can be seen

the six-membered ring. The observed equivalence of the protons of the methyl groups and of the methylene groups implies rapid oscillation of the Me_2C group about a mean position.

Inspection of the crystal packing shows that the intermolecular environment of the two nitroso-groups is quite different as, for example, only O(2) is involved in a short Se · · · O intermolecular contact. The near equality of the Se–O intramolecular distances is, therefore, probably a consequence of the intramolecular bonding rather than the crystal forces. While the difference in the Se–O distances is only slightly greater than the estimated standard deviations, the longer distance is to the oxygen atom involved in the intermolecular Se · · · O interaction.

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