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The Crystal and Molecular Structures of 2,5-Diaza-1,6-dioxa-6a-thiapentalene and its 6a-Selena and 6a-Tellura Analogs

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The crystal and molecular structures of 2,5-diaza-1,6-dioxa-6a-thiapentalene (I), 2,5-diaza-1,6-dioxa-6a-selenapentalene (II) and 2,5-diaza-1,6-dioxa-6a-tellurapentalene (III) have been determined by means of X-ray crystallographic methods.

Crystals of I are yellow, space group $P2_1/c$ with $Z=4$ in a unit cell of dimensions $a=6.939(5)$ Å, $b=7.000(5)$ Å, $c=11.013(5)$ Å and $\beta=110.83(7)°$.

Crystals of II are red, space group $P2_1/c$ with $Z=4$ in a unit cell of dimensions $a=6.987(1)$ Å, $b=7.000(2)$ Å, $c=11.379(2)$ Å and $\beta=110.93(2)°$.

Crystals of III are dark red, space group $P2_1/c$ with $Z=4$ in a unit cell of dimensions $a=9.605(4)$ Å, $b=5.006(2)$ Å, $c=12.202(3)$ Å and $\beta=112.94(3)°$.

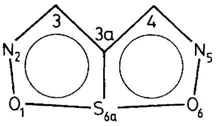
The structures were solved by direct methods (I), isomorphism (II), and Patterson methods (III), and refined by full-matrix least-squares techniques.

Within experimental errors, the three molecules are planar and symmetric about the respective C(3a)–X(6a) bonds (X = S,Se,Te).

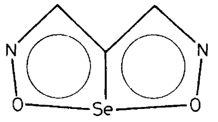
The average values for bond lengths are, O–S = 1.852(2) Å, C–S = 1.683(4) Å, O–N = 1.350(4) Å, C–N = 1.309(6) Å, and C–C = 1.407(5) Å for compound I, O–Se = 1.992(6) Å, C–Se = 1.827(8) Å, O–N = 1.33(1) Å, C–N = 1.33(1) Å, and C–C = 1.42(1) Å for compound II, and O–Te = 2.087(13) Å, C–Te = 1.980(15) Å, O–N = 1.37(3) Å, C–N = 1.39(3) Å, and C–C = 1.36(3) Å for compound III. The bond lengths in I and II have been corrected for libration.

Values of 1.19 Å for Se and 1.39 Å for Te are proposed for the covalent single bond radii of these elements in *cis* planar diselenide and ditelluride groups, respectively. The lengthening of the O–X (6a) bonds in compounds I–III relative to the corresponding sums of covalent radii then become 8.3 %, 7.6 % and 1.8 %, respectively, in agreement with results from ESCA studies and CNDO/2 calculations.

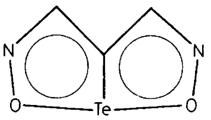
In crystals of I and II there are no intermolecular close contacts shorter than the corresponding van der Waals distances. The crystal structure of III is disordered (50–50) and a series of intermolecular close contacts is possible.



I



II



III

Compounds I, II and III are related to the 1,6,6a-trithiapentalenes, and the bonding in the O···X···O sequences may be described in terms of three-centre four-electron bonds.[1]

Possible differences between sulfur, selenium and tellurium with respect to such O···X···O bonding might be revealed through the molecular structures of I–III, and the present X-ray studies have therefore been carried out.

STRUCTURE DETERMINATION

Samples of compounds I, II and III were generously supplied by Vialle.[2]

Crystals of I from cyclohexane are yellow plates, which had to be kept in capillaries during the X-ray

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Table 1. Crystal data for compounds I, II and III.

	I $C_3H_2N_2O_2S$	II $C_3H_2N_2O_2Se$	III $C_3H_2N_2O_2Te$
F.W.	130.12	177.02	225.66
Colour	yellow	red	dark red
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$
a(Å)	6.939(5)	6.987(1)	9.605(4)
b(Å)	7.000(5)	7.000(2)	5.006(2)
c(Å)	11.013(5)	11.379(2)	12.202(3)
β(°)	110.83(7)	110.93(2)	112.94(3)
V(Å^3)	499.97	519.81	540.30
Z	4	4	4
D_m(g/cm^3)	1.71	2.26	2.74
D_x(g/cm^3)	1.729	2.262	2.774
μ(Mo$K\alpha$)(cm^{-1})	5.27	19.14	14.01

analyses in order to prevent sublimation. Recrystallization of II from a series of different solvents gave twinned crystals. One untwinned prismatic crystal was eventually found in a batch which had been recrystallized from ethanol by slow evaporation; the crystals of II are red. Crystals of III from benzene by slow evaporation are dark red needles elongated along *b*.

Crystal data for compounds I, II and III are given in Table 1. The crystal analyses are based on diffractometer data collected at 22 °C using Mo*K*α radiation. The intensity data for I and III have been collected on a paper-tape controlled Siemens AED diffractometer using niobium filtered radiation, and the intensity data for II were collected on a Nonius CAD4 computer-controlled diffractometer using graphite monochromatized radiation.

The intensities of the reflections for I and III were measured on the diffractometer by means of the five-value scan technique.[3] Reflections for which the net count was greater than two times the respective standard deviation in the net count were accepted as observed. With this criterion 630 independent reflections for I and 788 for II were accepted as observed in the θ-range $1-26°$; the corresponding numbers of observable reflections are 981 and 1064.

The intensity data for II were collected by the $\omega-2\theta$ scan technique. 2083 reflections were measured in the θ-range $1-33°$, and 1127 of these for which $I>2\sigma(I)$ were accepted as observed. The $0k\bar{l}$ as well as the $0kl$ reflections were measured and included in the data set.

The dimensions in axial directions of the crystals used for intensity measurements were $0.015 \times 0.52 \times 0.34$ mm for I, $0.5 \times 0.5 \times 0.5$ mm for II and $0.023 \times 0.42 \times 0.10$ mm for III. L*p* and absorption corrections were applied.[4]

The structure of compound I was solved by direct methods using a program written by Long[5] and the structural parameters were refined by full matrix least squares procedures (see, for example, Ref. 6).

Crystals of I and II are isomorphous and parameters from the refinement of I were used as a start for the refinement of II. The calculations on the latter compound were carried out by means of the X-RAY 76 program system.[7]

Compound III which has a disordered crystal structure was solved by Patterson methods. A careful search for reflections by means of film as well as diffractometer showed that no other reflections are present than those of space group $P2_1/c$. The disorder is therefore 50 − 50.

The Patterson map of III unambiguously showed that there are two tellurium positions per asymmetric unit, and this implies that only 50 % of the molecular positions given by the Patterson map can be occupied.

Due to heavy overlap caused by the disorder, one had to carry out the refinement very carefully. The computer programs used were those mentioned in Ref. 6.

For hydrogen the scattering factor curve given by Stewart *et al.*[8] was used. The other atomic scattering factors were in the case of compounds I and III taken from *International Tables*,[9] and in the case of compound II computed from numerical Hartree-

Table 2. Atomic coordinates (fractional) and temperature parameters (in Å^2) for compounds I and II. The expressions used are $\exp[-8\pi^2 U(\sin^2\theta/\lambda^2)]$ for hydrogen, and $\exp[-2\pi^2(h^2a^{*2}U_{11}...2hka^*b^*U_{12}+\cdots)]$ for the other atoms. The U_{ij}s have been multiplied by 10^4.

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(6a)	0.7659(2)	0.0937(1)	0.0747(1)	665(7)	428(5)	438(5)	−61(6)	249(5)	21(5)
O(1)	0.7752(4)	−0.1298(4)	−0.0105(2)	882(21)	695(21)	560(16)	−32(17)	411(16)	−150(15)
O(6)	0.7424(4)	0.2950(3)	0.1752(3)	942(22)	317(15)	680(18)	−13(16)	295(16)	−13(16)
N(2)	0.7626(6)	−0.2858(5)	0.0566(4)	796(26)	500(23)	733(25)	7(21)	350(21)	−154(21)
N(5)	0.7198(6)	0.2421(5)	0.2856(3)	973(31)	499(21)	620(24)	22(21)	346(22)	−71(22)
C(3)	0.7481(6)	−0.2410(5)	0.1663(4)	632(29)	386(22)	593(27)	12(21)	270(22)	−14(21)
C(3a)	0.7456(6)	−0.0446(4)	0.1925(3)	428(22)	378(19)	385(19)	−2(18)	163(16)	−23(16)
C(4)	0.7238(6)	0.0558(5)	0.2949(4)	752(29)	458(25)	428(22)	27(22)	291(21)	22(19)
H(3)	0.731(6)	−0.318(5)	0.225(3)	$U=0.078(13)$					
H(4)	0.712(5)	0.011(4)	0.369(3)	$U=0.056(12)$					
Se(6a)	0.7715(1)	0.1140(1)	0.0743(1)	482(5)	476(5)	287(4)	−60(6)	140(3)	30(5)
O(1)	0.7793(10)	−0.1274(10)	−0.0135(6)	741(43)	735(46)	535(34)	−109(44)	339(32)	−199(39)
O(6)	0.7476(10)	0.3084(8)	0.1949(5)	688(45)	354(30)	455(37)	−33(31)	104(34)	17(26)
N(2)	0.7593(12)	−0.2810(13)	0.0467(8)	581(52)	608(53)	689(61)	43(44)	219(49)	−297(45)
N(5)	0.7237(12)	0.2301(10)	0.2955(6)	702(57)	646(46)	514(49)	7(44)	226(44)	−90(39)
C(3)	0.7445(14)	−0.2405(13)	0.1580(10)	420(53)	331(45)	730(65)	50(43)	163(49)	−9(49)
C(3a)	0.7453(13)	−0.0489(10)	0.1911(7)	329(46)	303(38)	380(44)	2(33)	129(38)	4(32)
C(4)	0.7283(15)	0.0455(12)	0.2978(8)	602(61)	468(48)	322(42)	18(45)	170(43)	−4(37)
H(3)	0.768(15)	−0.345(12)	0.220(9)	$U=0.101(32)$					
H(4)	0.749(13)	−0.035(12)	0.373(7)	$U=0.085(27)$					

Table 3. Atomic coordinates (fractional) and temperature parameters (in Å^2) for compound III. The expressions used are $\exp[-2\pi^2(h^2a^{*2}U_{11}+\cdots 2hka^*b^*U_{12}+\cdots)]$ for tellurium and $\exp[-8\pi^2 U(\sin^2\theta/\lambda^2)]$ for the other atoms.

Molecule 1					Molecule 2			
Atom x	y	z	U		x	y	z	U
Te(6a) −0.0829(1)	0.2141(3)	0.0470(1)			0.5930(2)	−0.0879(3)	0.1200(1)	

Atom	x	y	z	U	x	y	z	U
Te(6a)	−0.0829(1)	0.2141(3)	0.0470(1)		0.5930(2)	−0.0879(3)	0.1200(1)	
O(1)	0.0717(19)	0.0757(34)	0.2126(13)	0.074(4)	0.6657(15)	−0.3464(28)	0.0193(14)	0.053(4)
O(6)	−0.1823(19)	0.2269(34)	−0.1377(19)	0.076(5)	0.4487(23)	0.2028(42)	0.1382(14)	0.068(5)
N(2)	0.1571(27)	−0.1327(54)	0.1982(22)	0.070(8)	0.6011(24)	−0.3164(40)	−0.0985(20)	0.055(5)
N(5)	−0.1302(40)	0.0437(67)	−0.1970(29)	0.103(12)	0.3478(28)	0.3318(45)	0.0379(27)	0.079(6)
C(3)	0.1358(35)	−0.1991(59)	0.0799(29)	0.093(9)	0.4949(33)	−0.1206(55)	−0.1357(23)	0.075(8)
C(3a)	0.0340(20)	−0.0561(39)	−0.0007(19)	0.056(5)	0.4717(19)	0.0248(34)	−0.0447(13)	0.041(4)
C(4)	−0.0170(29)	−0.1031(51)	−0.1245(23)	0.077(6)	0.3717(30)	0.2183(58)	−0.0633(26)	0.079(7)
H(3)	0.217(18)	−0.330(32)	0.068(14)	0.07(5)	0.432(18)	−0.079(31)	−0.215(14)	0.07(5)
H(4)	0.044(19)	−0.238(32)	−0.150(15)	0.06(5)	0.301(19)	0.306(32)	−0.124(15)	0.05(5)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Te(6a)$_1$	0.0502(7)	0.0608(10)	0.0657(9)	−0.0007(7)	−0.0005(7)	0.0166(6)
Te(6a)$_2$	0.0732(9)	0.0628(9)	0.0411(7)	0.0067(8)	−0.0026(7)	0.0204(6)

Fock wave functions.[10] The final R factors for compound I, II and III are 0.043, 0.061 and 0.049, respectively.

Final atomic coordinates and temperature parameters are listed in Tables 2 and 3. The structure factor lists are available on request.

Rigid body analyses have been carried out for compounds I and II according to the method of

Table 4. Bond lengths l (*Å*) in compounds I, II and III. The values l' have been corrected for rigid body libration. Standard deviations in parentheses.

	X=S l	X=Se l'	l	X=Te l'	Mol. 1 X=Te l	Mol. 2 X=Te l	Average X=Te
X(6a)–O(1)	1.838(3)	1.853	1.973(7)	1.987	2.105(14)	2.083(17)	2.094
X(6a)–O(6)	1.835(3)	1.850	1.982(6)	1.997	2.079(21)	2.081(22)	2.080
X(6a)–C(3a)	1.665(4)	1.683	1.810(8)	1.827	1.986(22)	1.973(14)	1.980
O(1)–N(2)	1.339(5)	1.353	1.31(1)	1.32	1.38(3)	1.34(3)	1.36
O(6)–N(5)	1.333(5)	1.347	1.33(1)	1.34	1.38(4)	1.39(3)	1.39)
C(3)–N(2)	1.286(7)	1.299	1.34(1)	1.35	1.42(4)	1.36(3)	1.39
C(4)–N(5)	1.308(5)	1.319	1.29(1)	1.30	1.33(4)	1.46(5)	1.40
C(3)–C(3a)	1.406(5)	1.418	1.39(1)	1.40	1.30(3)	1.42(4)	1.36
C(4)	1.382(6)	1.395	1.42(1)	1.44	1.41(3)	1.32(3)	1.37

Table 5. Bond angles (°) in compounds I, II and III. Standard deviations in parentheses.

i	*j*	*k*	X=S ∠*ijk*	X=Se ∠*ijk*	Mol. 1 X=Te ∠*ijk*	Mol. 2 X=Te ∠*ijk*	Average X=Te ∠*ijk*
O(1)	X(6a)	O(6)	171.5(2)	164.3(3)	154.8(7)	152.6(6)	153.7
O(1)	X(6a)	C(3a)	86.0(2)	82.0(3)	77.8(8)	77.1(7)	77.5
O(6)	X(6a)	C(3a)	85.8(2)	82.4(3)	77.0(8)	75.8(7)	76.4
X(6a)	O(1)	N(2)	113.0(2)	114.2(6)	110(2)	116(2)	113
X(6a)	O(6)	N(5)	113.7(2)	112.3(5)	116(2)	120(2)	118
O(1)	N(2)	C(3)	111.2(3)	112.4(8)	117(2)	115(2)	116
O(6)	N(5)	C(4)	110.0(3)	114.8(8)	113(3)	106(2)	110
N(2)	C(3)	C(3a)	116.7(3)	117.8(9)	114(3)	116(2)	115
N(5)	C(4)	C(3a)	116.2(3)	117.1(8)	118(3)	119(2)	119
X(6a)	C(3a)	C(3)	113.5(3)	113.6(7)	120(2)	116(3)	118
X(6a)	C(3a)	C(4)	113.8(3)	113.3(5)	115(2)	119(2)	117
C(3)	C(3a)	C(4)	132.7(3)	133.1(8)	124(3)	125(2)	125

Schomaker and Trueblood.[11] All the calculations were carried out on the UNIVAC 1110 of the University of Bergen.

DISCUSSION

Bond lengths and angles in compounds I, II and III are given in Tables 4 and 5, respectively.

The molecules are planar and, within the experimental error, symmetric about the respective C(3a)–X(6a) bonds. Average O–X(6a) bond lengths and O–X(6a)–O bond angles together with the sums of covalent radii for oxygen and sulfur, 1.71 Å,[13,14] oxygen and selenium, 1.83 Å,[13] and oxygen and tellurium, 2.03 Å[13], are given in Fig. 1. The standard deviations σ_{av} in the average bond lengths and angles are calculated from the individual standard deviations σ_i according to the formula

$$\sigma_{av}=(1/n)\left(\sum_{i=1}^{n}\sigma_i^2\right)^{\frac{1}{2}}$$

One notes from Fig. 1 that the lengthenings of the O–X(6a) bonds relative to the corresponding sums of covalent radii are 8.3, 8.9 and 2.8 %, respectively, for the sulfur, selenium and tellurium compounds. These lengthenings are seen to be smaller than those of 12.5, 10.2 and 10.4 % found for the analogous compounds IV, V and VI, respectively, *cf.* Fig. 2.[15,17]

ESCA studies and CNDO/2 calculations on compounds I–VI[18,19] show that the ground state potential wells for the O–X(6a)–O three-centre

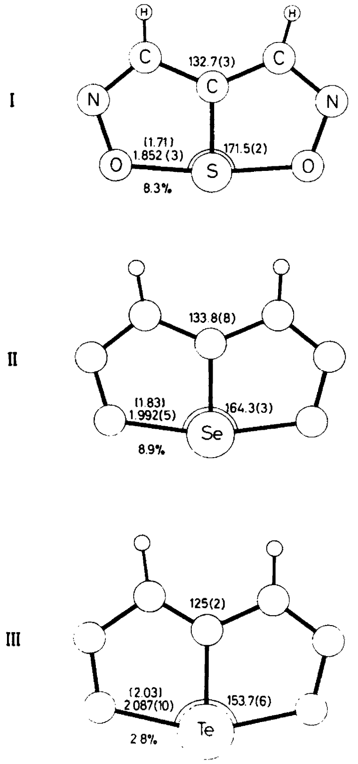

Fig. 1. ORTEP[12] drawings of the molecular structures of compounds I, II and III. The average O – X distances (Å), C – C – C and O – X – O angles (°) are given. The corresponding sums (Å) of covalent radii for O and X are shown in square brackets and the relative lengthening of the O – X bonds are given in percent.

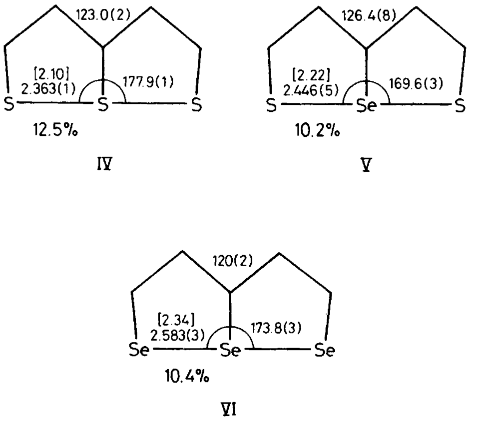

Fig. 2. The structures of 1,6,6a-trithiapentalene (IV) 1,6-dithia-6a-selenapentalene (V) and 1,6,6a-triselenapentalene (VI). The average S – S, S – Se, and Se – Se bond distances (Å) are given together with the corresponding sums of covalent radii (Å) in square brackets. Some bond angles (°), and the relative lengthenings (%) of S – S, S – Se and Se – Se bonds are also given.

bonds in compounds I – III are narrower than that of the S – S – S three-centre bond in compound IV. This implies that the O – X(6a) – O bonding in I – III is stronger than the S – S – S bonding in IV. The lengthening of the O – X(6a) bonds in compounds I – III should therefore, in agreement with the observations, be somewhat smaller than the lengthening of the S – S bonds in compound IV.

The ESCA studies and the CNDO/2 calculations show further that the widths of the ground state potential wells decrease from I – III and from IV – VI, and the bond lengthenings should therefore be expected to decrease in the same order. However, the calculated lengthenings do not decrease as expected, *cf.* Figs. 1 and 2, and a query might be put at the reference values used for the respective single bond lengths, as discussed below.

The length of the S – S single bond in a *cis* planar disulfide group, 2.10 Å,[14] is regarded as a relevant reference value when discussing sulfur – sulfur bonds in planar molecules, and a covalent radius of 1.05 Å for sulfur is accordingly used in the present paper. However, the covalent radii used above for selenium and tellurium do not refer to *cis* planar groups.

It has been shown, empirically and theoretically,[14,20] that the length of the S – S single bond in a disulfide group changes with the dihedral angle; 2.02 Å is a relevant length for the S – S single bond at 90° dihedral angle, and 2.10 Å is a relevant length at 0 and 180° dihedral angles. It is interesting to mention in this connection that the lengths of the S – S bonds in bis(2-pyrimidyl) disulfide at C – S – S – C dihedral angles of 82.5°,[21] 84.6°[22] and 180°,[22] are found to be 2.016(1), 2.019(1) and 2.113(1) Å, respectively.

Calculations on diselenide and ditelluride groups show similar variations of Se – Se and Te – Te bonds with dihedral angle,[20] and the range of the bond length variations are, according to the calculations, almost the same for the Se – Se and Te – Te bonds as for the S – S bond.

A more realistic value for the Se – Se single bond

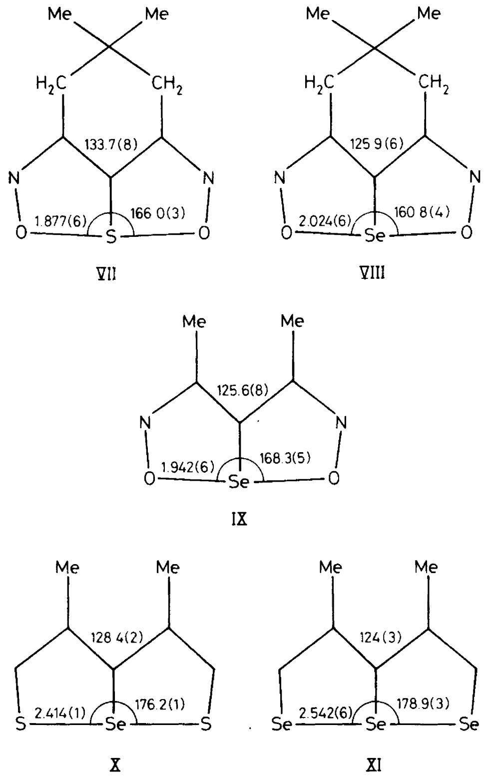

Fig. 3. The structures of derivatives of compounds I, II, V, and VI showing average O – S, O – Se, S – Se, and Se – Se bond lengths (Å) and some bond angles (°).

length in *cis* planar diselenide groups may be obtained if one compares the average Se – Se bond length in Se_8, 2.334(5) Å,[23] with the average bond length in S_8, 2.048(2) Å.[24] The latter is seen to be 0.05 Å shorter than the S – S single bond in *cis* planar groups, and, if this difference is added to the former, one arrives at a value of 2.38 Å for the Se – Se single bond length in *cis* planar diselenide groups. A covalent single bond radius of 1.19 Å may therefore be proposed for Se in such groups. Similarly 1.39 Å may be proposed as a more realistic value than 1.37 Å for the covalent radius of Te in *cis* planar ditelluride groups; the latter value is representative for dihedral angles about 90°.[20] It is realized that the same conclusions have been reached by Sæthre *et al.* from a different view point.[18]

If the values 1.19 and 1.39 Å are used for the covalent radii of Se and Te, respectively, the lengthenings of S – S, S – Se and Se – Se bonds in compounds IV – VI become 12.5, 9.3 and 8.5 %, and the lengthenings of O – S, O – Se and O – Te bonds in compounds I – III become 8.3, 7.6 and 1.8 %, respectively. This agrees with the mentioned ESCA studies and CNDO/2 calculations.

Structures of three derivatives of compounds I and II have been studied earlier, *cf.* Fig. 3.[25-27] One notes that the O – X(6a) bond lengths in these compounds are somewhat different from those in compounds I and II. This illustrates, in fact, to which extent substituents may influence the bonding in compounds I and II. Thus the substituents in VII and VIII have caused lengthenings of the respective O – X(6a) bonds, while the methyl groups in IX have shortened the O – Se(6a) bonds there relative to those in II. The latter effect is also seen if one compares the S – Se and Se – Se bond lengths in X[28] and XI[29] with those in V and VI, respectively, *cf.* Figs. 2 and 3.

Comparison with 1,6,6a-trithiapentalene. A survey of bond lengths in 1,6,6a-trithiapentalenes has been reported.[15] This survey shows that the terminal C(2) – C(3) and C(4) – C(5) bonds are, as a rule, shorter and has more π-character than the central C(3) – C(3a) and C(4) – C(3a) bonds. The same structural feature is found for compounds I and II. Thus the average value of the terminal C – N bonds in I is 1.309(6) Å and in II 1.33(1) Å, and the average values of the central C – C bonds in I and II are 1.407(6) and 1.42(1) Å, respectively, *cf.* Table 4. The C – C and C – N bond lengths in III are not considered accurate enough to be included in this discussion.

The mentioned survey shows further that the central C(3a) – S(6a) bonds in 1,6,6a-trithiapentalenes have a rather constant length of about 1.75 Å, and the terminal C(2) – S(1) and C(5) – S(6) bonds are usually found to be somewhat shorter, about 1.70 Å.

Accepted values for C – S single and double bond lengths are 1.82 and 1.61 Å, respectively.[33] Thus, as a rule, the terminal C – S bonds in 1,6,6a-trithiapentalenes possess more double bond character than the central ones.

The central C(3a) – S(6a) bond in I, 1.683(4) Å, is found to be significantly shorter than that in 1,6,6a-trithiapentalenes. The same applies to the C(3a) – Se(6a) bond in II, 1.827(8) Å, when compared with the average C(3a) – Se(6a) bond length of 1.91(1) Å found for 1,6,6a-triselenapentalenes and 1,6-dithia-6a-selenapentalenes.[16,17,28-32] Accepted

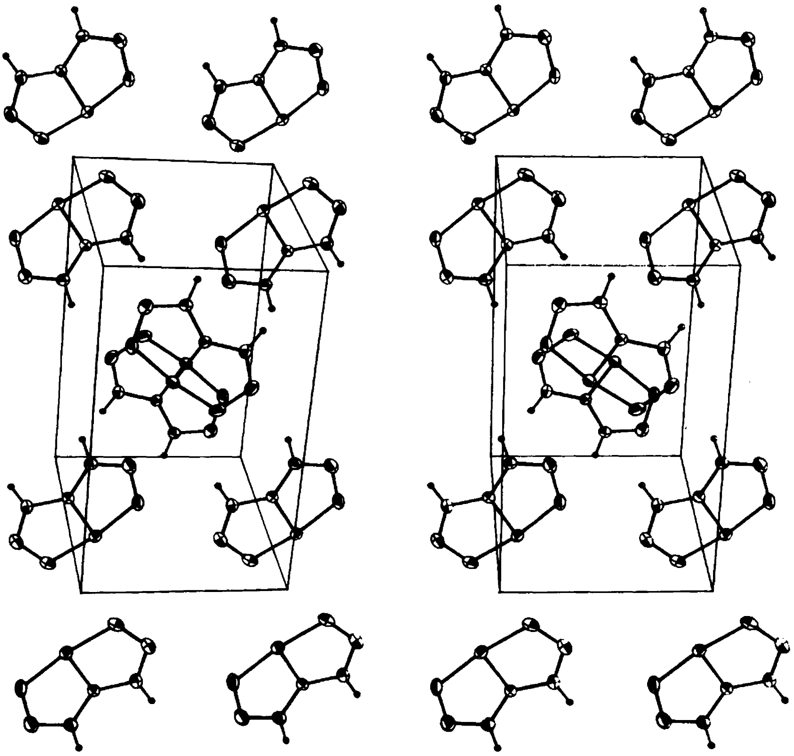

Fig. 4. A stereo view of the crystal structure of I.

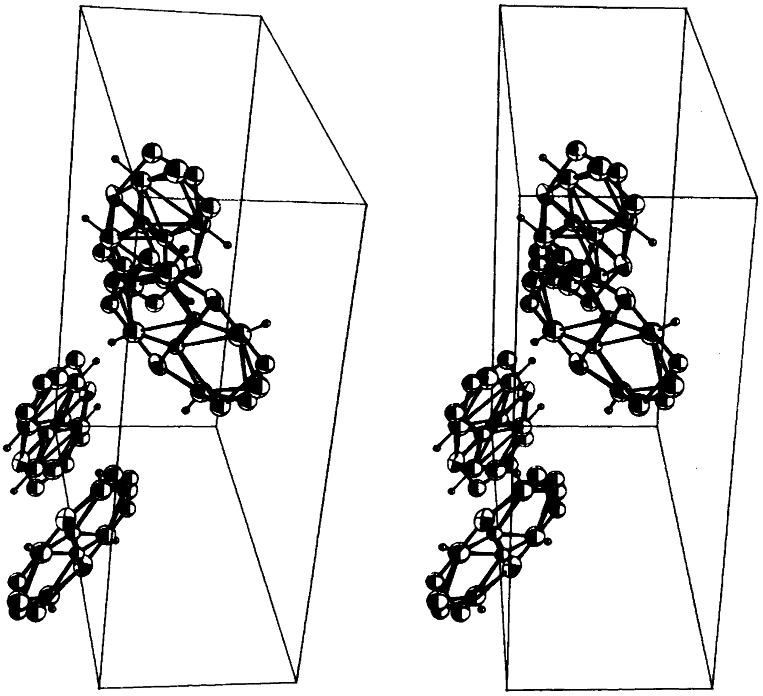

Fig. 5. A stereo view of the crystal structure of III showing the disorder.

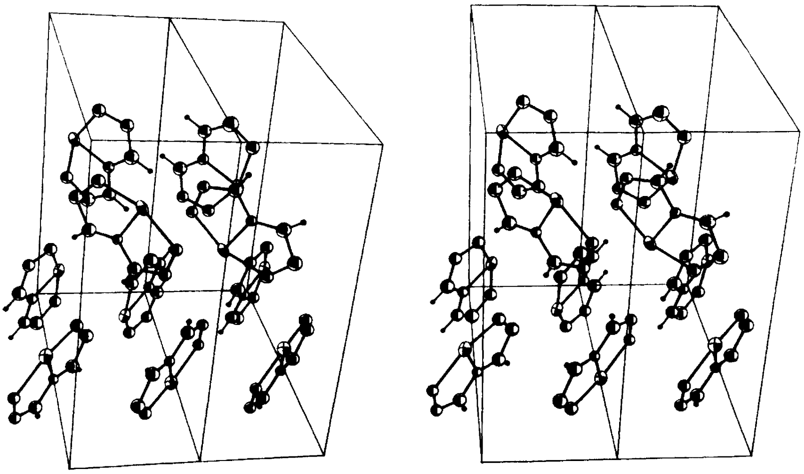

Fig. 6. A stereo view of the crystal structure of III showing an example of the real crystal packing.

values for C–Se single and double bond lengths are 1.94 and 1.74 Å, respectively.[13]

The average N–O bond length is 1.350(5) Å in I and 1.33(1) Å in II, *cf.* Table 4. Accepted lengths for N–O single and double bonds are 1.46 and 1.25 Å, respectively.[13] Thus one may conclude that the double bond characters of the N–O bonds in I and II are about the same as those of the C(3a)–X(6a) bonds in the respective molecules.

Crystal structures. A stereo view of the crystal structure of I is given in Fig. 4. The crystal structure of II is similar since crystals of the two compounds are isomorphous. There are no intermolecular contacts closer than corresponding van der Waals distance in crystals of I and II.

The crystal structure of compound III is disordered. Fig. 5 illustrates the disorder, and Fig. 6 gives one example of real crystal packing. There are a series of possible intermolecular close contacts in the crystal structure of III. These are, two possible Te···Te contacts of 3.67 and 3.86 Å, respectively, five possible Te···O contacts in the range 3.19–3.50 Å and seven possible Te···N contacts in the range 3.26–3.60 Å.

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