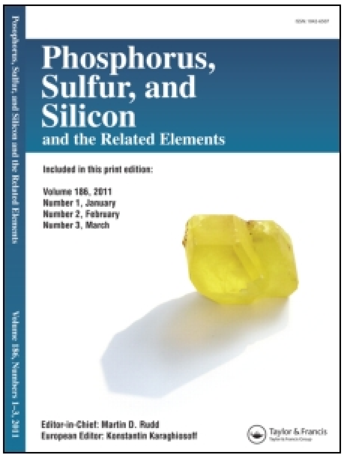

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CRYSTAL STRUCTURES OF A TETRAOXY SPIROCYCLIC SELENURANE AND TELLURANE. LONE PAIR EFFECTS

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CRYSTAL STRUCTURES OF A TETRAOXY SPIROCYCLIC SELENURANE AND TELLURANE. LONE PAIR EFFECTS[1]

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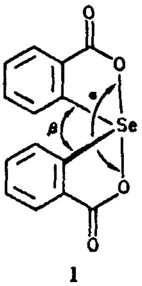
(Received December 15, 1980)

The crystal structures of the tetraoxy spirocyclic selenurane $(C_6H_{12}O_2)_2Se$ (**3**) and corresponding tellurane $(C_6H_{12}O_2)_2Te$ (**4**) have been determined by single-crystal X-ray diffraction analysis. Both compounds crystallize in the monoclinic space group $C2/c$ with $Z = 4$ and with the central atom on a crystallographic twofold axis. For **3**, $a = 10.442$ (6) Å, $b = 10.545$ (5) Å, $c = 13.910$ (3) Å, and $\beta = 110.27$ (3)°. For **4**, $a = 19.108$ (7) Å, $b = 10.469$ (4) Å, $c = 7.705$ (2) Å, and $\beta = 97.83°$. Data for both compounds were collected on an Enraf-Nonius CAD4 diffractometer out to a maximum $2\theta_{MoK\bar{\alpha}}$ of 50°. Full-matrix least-squares refinement led to $R = 0.042$ and $R_w = 0.055$ for **3** and $R = 0.034$ and $R_w = 0.042$ for **4**. Both exist in a trigonal-bipyramidal structure distorted in compliance with the presence of an equatorially positioned lone electron pair. In the Te compound intermolecular Te—O contacts indicate a degree of polymerization making the Te atoms pseudo six-coordinated. No such contact is observed in the Se moiety.

INTRODUCTION

Although the molecular structures of spirocyclic phosphoranes have been extensively studied[2] and those of spirocyclic sulfuranes[3] studied to a lesser extent, spirocyclic selenuranes[4] have received little attention and related telluranes apparently none at all. The structure of 3,3′-spirobi(3-selenaphthalide) (**1**) is known[4] (for this and other structures to follow, the axial angle α and equatorial angle β refer to the angles as designated in **1**). Like most cyclic sulfuranes,[3] the structure of the selenurane **1** shows the axial angle O—Se—O and equatorial angle C—Se—C bent away from the fifth coordination site, an equatorial site presumed to be occupied



1
O_{ax}-Se-O_{ax} = 172.4 (3)°
C_{eq}-Se-C_{eq} = 101.0 (3)°

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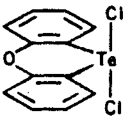
by a lone electron pair. This contrasts with the structures of cyclic phosphoranes which show the axial and equatorial angles bent in mutually opposing directions (i.e., along the Berry coordinate[5]) and yield a range of structures between the idealized trigonal bipyramid and square (or rectangular) pyramid.[2,6]

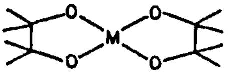
A number of monocyclic tellurane structures has been reported[7] which have axial and equatorial angles bent in the same direction as that for the selenurane **1**. Tellurane **2**[8] is representative of this class.



2

Cl_{ax}-Te-Cl_{ax} = 176.8 (1)°
C_{eq}-Te-C_{eq} = 90.7 (3)°

In order to strengthen the structural basis for determining pentacoordinate principles[2] applicable to group 6 derivatives, we carried out a single-crystal X-ray study of the similarly formulated spirocyclic selenurane **3** and the spirocyclic tellurane **4**. Temperature-dependent ^{13}C and ^{1}H NMR data support trigonal-bipyramidal ground-site structures for both **3**[9] and **4**[10] near −110°C. The onset of intramolecular ligand exchange, as indicated by the presence of nonequivalent pairs of methyl groups and a single type of ring carbon atom, occurs at higher temperatures. An exchange barrier of 8 kcal/mol was obtained for **3**,[9] indicating fluxional behavior for these substances.



(– = CH_3)
3, M = Se
4, M = Te

EXPERIMENTAL

Samples suitable for X-ray studies were kindly supplied by Professor D. B. Denney, Rutgers University.

Space Group Determination for **3** *and* **4**. Crystals of both **3** and **4** are colorless but become yellow on prolonged exposure to the X-ray beam, with an accompanying loss in diffracted intensity. For each compound it was possible, by starting with a fresh crystal, to obtain a data set before the onset of degradation. All studies were done with crystals mounted in sealed capillaries, and it is not known if degradation would have been more rapid otherwise. For both compounds, preliminary investigations using an Enraf-Nonius CAD 4 automated diffractometer and graphite-monochromated molybdenum radiation (fine-focus tube, 45 kV, 20 mA, takeoff angle = 3.1°, $\lambda(K\alpha_1)$ = 0.709 30 Å, $\lambda(K\alpha_2)$ = 0.713 59 Å) showed monoclinic (2/*m*) symmetry. The observed extinctions *hkl*, $h + k = 2n + 1$, and *h*0*l*, $l = 2n + 1$, indicated either space group *C*2/*c* or *Cc*.

For **3**, the lattice constants as determined by the least-squares refinement of the diffraction geometry for 25 reflections having $10.13° \leq \theta_{MoK\bar{\alpha}} \leq 16.65°$ and measured at an ambient laboratory temperature of 23 ± 2°C are a = 10.442 (6) Å, b = 10.545 (5) Å, c = 13.910 (3) Å, and β = 110.27 (3)°. A unit cell content of four molecules gives a calculated volume of 21.1 Å^3/nonhydrogen atom, which falls in the range expected for such molecules. For **4**, on the basis of 25 reflections having $10.73° \leq \theta_{MoK\bar{\alpha}} \leq 15.43°$, the lattice constants are a = 19.108 (7) Å, b = 10.469 (4) Å, c = 7.705 (2) Å, and β = 97.83°, with a unit cell content of four molecules giving a calculated volume of 22.5 Å^3/nonhydrogen atom. In each case, the assignment of $Z = 4$ was confirmed by successful solution and refinement of the structures in the space group $C2/c$ (C_{2h}^6, No. 15),[11] with the central atom on a crystallographic twofold axis and half a molecule in the asymmetric unit.

Data Collection for **3**. A colorless crystal having dimensions of 0.20 × 0.25 × 0.35 mm was mounted in a sealed thin-walled glass capillary for data collection. Data were collected with use of the θ-2θ scan mode with a θ scan range of $(0.82 + 0.35 \tan \theta)°$ centered about the calculated Mo $K\bar{\alpha}$ peak position. The scan range was actually extended an extra 25% on either side of the aforementioned limits for the measurement of background radiation. The scan rates varied from 1.3 to 6.7°/min, the rate to be used for each reflection having been determined by a prescan. The intensity, I, for each reflection is then given by $I = (\mathrm{FF}/S)][P - 2(B_1 + B_2)]$, where P are the counts accumulated during the peak scan, B_1 and B_2 are the left and right background counts, S is an integer which is inversely proportional to the scan rate, and FF is either unity or a multiplier to account for the occasional attenuation of the diffracted beam. The standard deviations in the intensities, σ_I, were computed as $\sigma_I^2 = (\mathrm{FF}^2/S^2)[P + 4(\mathrm{B1} + \mathrm{B2})] + 0.002I^2$. A total of 1263 independent reflections $(+h, +k, \pm l)$ having $2° \leq 2\theta_{\mathrm{MoK}\bar{\alpha}} \leq 50°$ was measured. Six standard reflections, monitored after every 6000 s of X-ray exposure time, gave no indication of crystal deterioration or loss of alignment. No corrections were made for absorption ($\mu_{\mathrm{MoK}\bar{\alpha}} = 2.813\ \mathrm{mm}^{-1}$), and the intensities were reduced to relative amplitudes by means of standard Lorentz and polarization corrections, including corrections for the monochromator.

Data Collection for **4**. Experimental conditions were the same as described for **3** unless otherwise noted. The colorless crystal used for data collection had dimensions of 0.14 × 0.15 × 0.27 mm. The scan range used was $(0.88 + 0.35 \tan \theta)$, with scan rates that varied between 0.74 and 4.0°/min. A total of 1343 independent reflections was measured, and five intensity controls were monitored. No corrections were made for absorption ($\mu_{\mathrm{MoK}\bar{\alpha}} = 2.813\ \mathrm{mm}^{-1}$).

Solution and Refinement for **3**. Initial coordinates for the nine independent nonhydrogen atoms were obtained by direct methods (MULTAN). Isotropic unit-weighted full-matrix least-squares refinement[12] of the structural parameters for these nine atoms and a scale factor gave a conventional residual $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ of 0.064 and a weighted residual $R_w = \{\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2\}^{1/2}$ of 0.077 for the 739 reflections having $I \geq 2\sigma_I$ and $(\sin \theta)/\lambda \leq 0.52$. Anisotropic refinement then gave $R = 0.055$ and $R_w = 0.069$. Peaks corresponding to the 12 independent hydrogen atoms were found on a difference Fourier synthesis run at this point. However, some of these were poorly resolved and had poor geometry which was not improved upon refinement. Accordingly, the coordinates of the stronger, better resolved peaks (one from each methyl group) were used to calculate coordinates giving reasonable geometry for the remaining H atoms. All hydrogen atoms were then included in subsequent refinement as fixed isotropic contributions. Refinement including the hydrogen atoms and using variable weight ($w^{1/2} = 2F_oLp/\sigma_I$) then gave $R = 0.038$ and $R_w = 0.052$.

The final cycles of refinement included the high-angle data and led to the final values of $R = 0.042$, $R_w = 0.055$, and GOF[13] = 1.554 for the 1052 reflections having $I \geq 2\sigma_I$ and $2° \leq 2\theta_{\mathrm{MoK}\bar{\alpha}} \leq 50°$. During the final cycle of refinement the largest shift in any parameter was 0.001 times its estimated standard deviation. The only peaks of any consequences on a final difference Fourier synthesis (0.743 and 0.694 e/Å^3) were in the immediate vicinity of the Se atom.

Solution and Refinement for **4**. Initial coordinates for the Te atom were deduced from a Patterson synthesis, while initial coordinates for the remaining eight independent nonhydrogen atoms were obtained by standard Fourier difference techniques. Isotropic unit-weighted refinement of the structural parameters for the nine independent nonhydrogen atoms and a scale factor gave $R = 0.063$ and $R_w = 0.074$ for the 837 reflections having $I \geq 3\sigma_I$ and $(\sin \theta)/\lambda \leq 0.52$. Anisotropic refinement then gave $R = 0.041$ and $R_w = 0.055$. Initial coordinates for the hydrogen atoms were then obtained from a difference Fourier synthesis, but, as in the case of **3**, the hydrogen atom geometries were poor before and after refinement. Therefore, coordinates for the hydrogen atoms were obtained as described for **3**. Inclusion of the hydrogen atoms as fixed isotropic scatterers in subsequent refinement using variable weights gave $R = 0.033$ and $R_w = 0.042$ for the 848 low-angle reflections having $I \geq 2\sigma_I$.

Inclusion of the high-angle data in the refinement led to the final values of $R = 0.034$, $R_w = 0.042$, and GOF[14] = 1.500 for the 1243 reflections having $I \geq 2\sigma_I$ and $2° \leq 2\theta_{\mathrm{MoK}\bar{\alpha}} \leq 50°$. During the final cycle of refinement the largest Δ/σ was 0.01. The only peaks of any consequence on a final difference Fourier synthesis (0.776, 0.611, and 0.530 e/Å^3) were in the immediate vicinity of the Te atom.

Computations were done on a CDC Cyber-175 computer with use of MULTAN, the direct methods program by Main, Germain, and Woolfson, LINEX, a modification of the Busing and Levy full-matrix least-squares program, ORFLS, Johnson's thermal ellipsoid plot program, ORTEP, the Oak Ridge Fortran function and error program, ORFFE, Zalkin's Fourier program, FORDAP, and several locally written programs.

RESULTS AND DISCUSSION

Figure 1 shows the molecular geometry and the atom labeling scheme for the spirocyclic selenurane **3**. Atomic coordinates are given in Tables I and II. For the corresponding tellurane, **4**, the analogous information is presented in Figure 2 and in Tables III and IV. For both compounds, bond lengths and angles for nonhydrogen atoms are given in Table V. Anisotropic thermal parameters and bond lengths and angles involving hydrogen atoms are available as supplementary material.

The similarity in the molecular geometry of the two compounds can be seen readily by comparing Figures 1 and 2. The geometry about the central atom in both cases is, to a first approximation, trigonal bipyramidal, with O1 and O1′ assuming axial positions and O2, O2′, and presumably a lone pair occupying equatorial positions. This general structural representation found in the solid state agrees with the structures supported for **3** and **4** in solution from low-temperature NMR data.[9,10] As has been observed for spirocyclic phosphoranes,[2a,15] the axially bonded

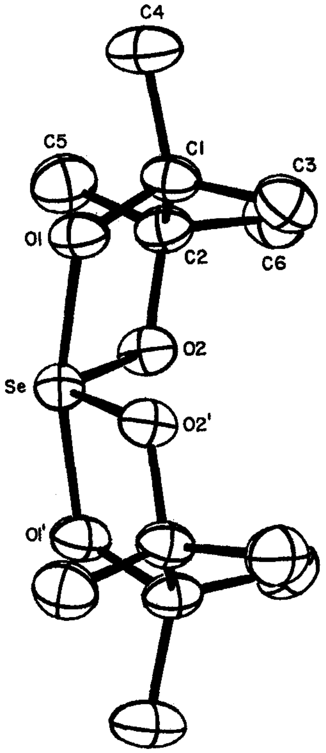


FIGURE 1 ORTEP plot of the $(C_6H_{12}O_2)_2Se$ molecule (**3**) with thermal ellipsoids shown at the 50% probability level. Primed atoms are related to unprimed ones by the crystallographic twofold axis through the Se atom. Hydrogen atoms are omitted for purposes of clarity.

TABLE I

Atomic Coordinates in Crystalline $(C_6H_{12}O_2)_2Se$ (3)[a]

atom type[b]	10^4x	10^4y	10^4z
Se	0[c]	6286.3 (7)	2500[c]
O1	720 (3)	6067 (3)	3919 (3)
O2	1375 (3)	5319 (3)	2457 (3)
C1	1691 (5)	5052 (5)	4236 (4)
C2	2449 (5)	5129 (5)	3442 (4)
C3	952 (6)	3790 (5)	4192 (4)
C4	2626 (6)	5336 (6)	5335 (4)
C5	3430 (5)	6238 (5)	3626 (4)
C6	3162 (6)	3902 (5)	3337 (5)

[a] Entries in parentheses are estimated standard deviations in the last significant figure. [b] Atoms are labeled to agree with Figure 1. [c] Fixed.

TABLE II

Fixed Parameters for Hydrogen Atoms in Crystalline $(C_6H_{12}O_2)_2Se$ (3)[a]

atom type[b]	10^4x	10^4y	10^4z
H31	500	3500	3400
H32	248	3910	4495
H33	1613	3158	4580
H41	3430	4756	5515
H42	3000	6250	5400
H43	2147	5211	5805
H51	3645	6395	3014
H52	4256	6033	4204
H53	3000	7000	3800
H61	3709	3604	4033
H62	2500	3250	3000
H63	3759	4068	2951

[a] Isotropic thermal parameters were fixed at 5 A^2. [b] H atoms are named for the C atom to which they are bonded, where H31, H32, and H33 are bonded to C3, etc.

atoms are at a further distance from the central atom than are the equatorially bonded atoms. For **3**, Se—O_{ax} = 1.867 (3) Å and Se—O_{eq} = 1.779 (3) Å, while for **4**, Te—O_{ax} = 2.007 (3) Å and Te—O_{eq} = 1.941 (3) Å.

The conformations of the 5-membered rings in the selenurane **3** and tellurane **4** are also very similar to one another and are most likely due to mitigation of crowding by staggering the methyl groups with respect to the C1—C2 bond. The puckering in the 5-membered rings can be described as a twisting of the C1—C2 portion out of the plane of the three remaining ring atoms (see planes III of Table VI).

In the idealized trigonal-bipyramidal geometry the ax—M—ax angle would be 180° and the eq—M—eq angle would be 120°. For **3** these values are 165.8 (2) and 110.0 (2)°, and for **4** they are 153.5 (2) and 105.6 (2)°, indicating that the distortions away from idealized geometry are not along the coordinate connecting the trigonal bipyramid to the rectangular pyramid but along a projection of this C_{2v} coordinate away from the rectangular pyramid.

For the selenurane **3** it may be argued that the observed distortion is exclusively a result of repulsion by the lone pair. For the tellurane **4** where the distortion is

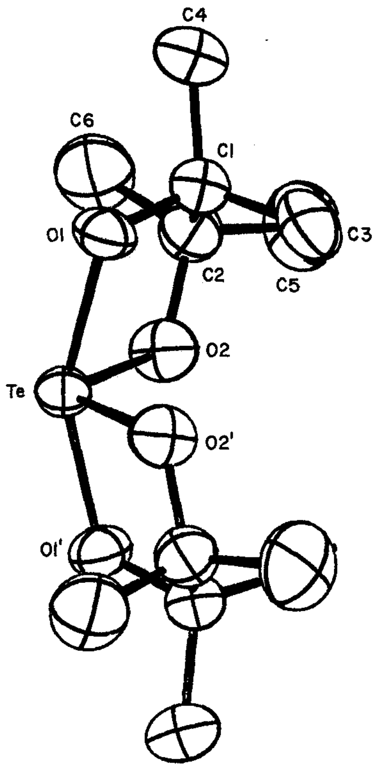


FIGURE 2 ORTEP plot of the $(C_6H_{12}O_2)_2Te$ molecule (4) with thermal ellipsoids shown at the 50% probability level. Primed atoms are related to unprimed ones by the crystallographic twofold axis through the Te atom. Hydrogen atoms are omitted for purposes of clarity.

TABLE III

Atomic Coordinates in Crystalline $(C_6H_{12}O_2)_2Te$ (4)[a]

atom type[b]	10^4x	10^4y	10^4z
Te	0[c]	809.1 (3)	2500[c]
O1	394 (2)	1248 (3)	290 (4)
O2	765 (2)	1930 (3)	3460 (4)
C1	860 (3)	2318 (5)	429 (7)
C2	1279 (3)	2214 (5)	2307 (8)
C3	437 (4)	3545 (6)	155 (9)
C4	1341 (4)	2195 (6)	−1013 (11)
C5	1628 (4)	3464 (7)	2989 (12)
C6	1820 (3)	1129 (7)	2467 (12)

[a] Entries in parentheses are estimated standard deviations in the last significant figure. [b] Atoms are labeled to agree with Figure 2. [c] Fixed.

even greater, intermolecular Te—O contacts of 3.064 (3) Å (compared to the van der Waals sum of 3.60 Å[16]) indicate that a bonding interaction may be responsible. This interaction is illustrated in Figure 3 where it can be seen that each axial oxygen atom is in contact with the Te atom of an adjacent molecule, while each Te atom

TABLE IV

Fixed Parameters for Hydrogen Atoms in Crystalline $(C_6H_{12}O_2)_2Te$ (4)[a]

atom type[b]	10^4x	10^4y	10^4z
H31	705	4210	−135
H32	238	3762	1225
H33	46	3415	−806
H41	1596	2997	−1109
H42	1640	1551	−786
H43	1037	2035	−2146
H51	1260	4155	2989
H52	1977	3733	2236
H53	1866	3344	4188
H61	2224	1406	1837
H62	1603	353	1927
H63	1990	968	3703

[a] Isotropic thermal parameters were fixed at 6 Å^2. [b] See footnote *b* of Table II.

TABLE V

Bond Lengths (Å) and Bond Angles (Deg) for $(C_6H_{12}O_2)_2Se$ (3) and $(C_6H_{12}O_2)_2Te$ (4)[a]

	bond length	
atom type[b]	M = Se	M = Te
M–O1	1.867 (3)	2.007 (3)
M–O2	1.779 (3)	1.941 (3)
O1–C1	1.435 (6)	1.426 (6)
O2–C2	1.453 (5)	1.442 (6)
C1–C2	1.566 (7)	1.560 (8)
C1–C3	1.528 (8)	1.517 (8)
C1–C4	1.531 (6)	1.541 (7)
C2–C5	1.517 (7)	1.529 (8)
C2–C6	1.527 (7)	1.528 (8)

	bond angle	
type	M = Se	M = Te
O1–M–O1′	165.8 (2)	153.5 (2)
O2–M–O2′	110.0 (2)	105.6 (2)
O1–M–O2	86.0 (1)	80.5 (1)
O1–M–O2′	85.9 (2)	83.6 (1)
M–O1–C1	113.6 (3)	114.8 (3)
M–O2–C2	114.6 (3)	115.5 (3)
O1–C1–C2	102.7 (4)	104.6 (4)
O2–C2–C1	104.9 (4)	106.2 (4)
O1–C1–C3	110.1 (4)	109.9 (4)
O1–C1–C4	107.4 (4)	107.9 (5)
C3–C1–C4	110.5 (4)	109.2 (5)
C3–C1–C2	112.8 (4)	112.6 (5)
C4–C1–C2	112.8 (4)	112.4 (5)
O2–C2–C5	108.0 (4)	105.5 (5)
O2–C2–C6	105.6 (4)	107.8 (5)
C5–C2–C6	110.3 (4)	110.5 (5)
C6–C2–C1	113.9 (4)	112.5 (5)
C5–C2–C1	113.4 (4)	113.8 (5)

[a] See footnote *a* of Table I. [b] Atoms are labeled to agree with Figures 1 and 2, where primed atoms go into unprimed ones by the molecular twofold axis.

TABLE VI

Deviations (Å) from Selected Least-Squares Mean Planes for $(C_6H_{12}O_2)_2Se$ (3) and $(C_6H_{12}O_2)_2Te$ (4)[a]

atoms	I	II	III	IV
Se	0.0	0.0	0.0	−0.117
O1	0.0		0.0	0.139
O1′	0.0		(−0.377)	
O2		0.0	0.0	0.018
O2′		0.0	(−1.663)	
C1			(−0.345)	−0.038
C2			(0.312)	(0.543)

atoms	I	II	III	IV	V
Te	0.0	0.0	0.0	0.101	0.0
O1	0.0	(−1.952)	0.0	−0.133	(−1.319)
O1′	0.0	(1.952)	(−0.772)		(1.319)
O2	(1.545)	0.0	0.0	−0.047	(−1.176)
O2′	(−1.545)	0.0	(−1.844)		(1.176)
C1			(−0.338)	(−0.610)	
C2			(0.310)	0.079	
O1″	(1.471)	(1.658)			0.0
O1‴	(−1.471)	(−1.658)			0.0

[a] Entries in parentheses are for atoms not included in the calculation of the plane. For 3, dihedral angle between planes I and II = 89.97°. For 4, dihedral angles between planes I and II = 88.00°, planes I and V = 42.46°, and planes II and V = 49.54°.

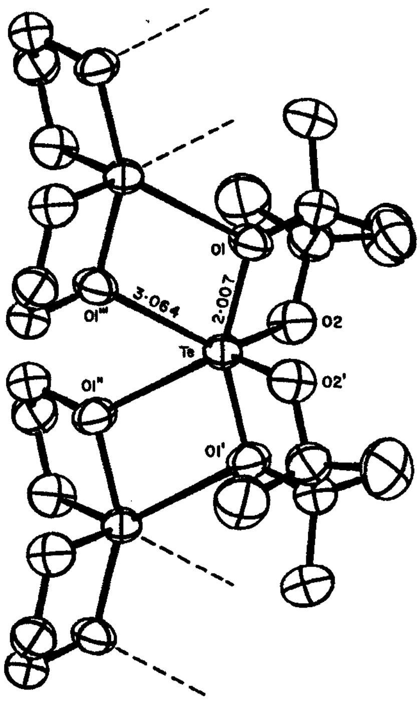
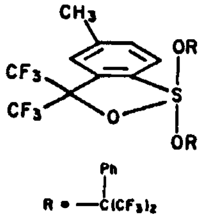


FIGURE 3 ORTEP plot showing the nature of the polymerization in crystalline $(C_6H_{12}O_2)_2Te$ (4). Methyl groups have been omitted from two of the three molecules for purposes of clarity. The propagation of the polymeric chain is indicated by the dashed lines.

is in contact with two oxygen atoms, one from each of two twofold related adjacent molecules. The two Te—O contacts make an angle of 90.7° with each other and are directed on each side of the assumed lone electron pair but not in the equatorial plane. The oxygen atom displacements from the equatorial plane are given in Table VI (plane V). This interaction thus results in a polymeric chain. For **3**, where the closest intermolecular contacts to the Se atom involve methyl groups of the adjacent molecules, no such tendency to polymerize is observed.

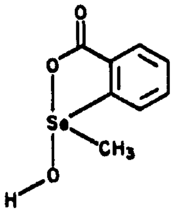
In the tellurane **2**, like **4**, intermolecular contacts influence the primary geometry.[8] In this case, a distorted trigonal bipyramid results as the intermolecular contacts, which are symmetrically placed in the equatorial plane about the lone pair, provide discrete tetrameric clusters.[8] The presence of the ring system in diequatorial positions instead of the axial-equatorial orientation is most probably associated with the low ring strain[2] energy in this disposition for pentacoordinated structures containing a lone electron pair.[17,18] It is noted that the structure of the monocyclic sulfurane **5**[19] shows a five-membered ring located diequatorially. For **5**, like **2**, the ligand preference rule[20] is met which states that the most electronegative ligands occupy axial positions. A steric effect associated with the large axial OR groups may also act to stabilize this structure. The structures of the spirocyclic selenuranes **1**[4] and **3** are similar and not too different from the structure assumed by the monocyclic selenurane **6**.[21] Here, the equatorial-equatorial ring orientation is precluded owing to the accompanying placement of a methyl group in an axial site.



5

$O_{ax}\text{-S-}O_{ax} = 172.0\ (4)^\circ$

$C_{eq}\text{-S-}O_{eq} = 95.0\ (6)^\circ$



6

$O_{ax}\text{-Se-}O_{ax} = 169.9\ (2)^\circ$

$C_{eq}\text{-Se-}C_{eq} = 98.8\ (2)^\circ$

The apparent ease of accommodation of small-membered rings in diequatorial positions of a trigonal bipyramid for group 6 pseudopentacoordinate derivatives suggests that the relative ligand apicophilicity is a dominant structural determinant[2b] in contrast to the more equal role that ring strain and ligand apicophilicity play in influencing structure for phosphoranes.

The axial angle, O—Se—O, in both **1** and **3** is about 5° smaller than the O—S—O angle in related cyclic sulfuranes.[2b,3] This presumably is partly a consequence of the longer Se—O bond over the S—O bond in the five-membered rings. However, changing the equatorial ring atom electronegativity from carbon to oxygen in the five-membered ring sulfuranes leads to a lowering of the equatorial angle; cf. the C—S—C angle of ~108° with an O—S—O angle of ~105°. In the selenuranes **1**[4] and **3**, the opposite is present (for **1** the C—Se—C angle is 101.0 (3)°; for **3** the O—Se—O angle is 110.0 (2)°). Reduced bond electron pair repulsion effects[22] with the use of oxygen atoms would give a reduction in the equatorial angle as observed for the cyclic sulfuranes.[2b,3] In Table VI, plane III shows that for **3** the displacement of C2 attached to the equatorial oxygen atom O2 (0.312 Å in the direction of the equatorially positioned lone pair) may be responsible for the 110.0° O—Se—O equatorial angle. This carbon atom displacement, as commented earlier, seems to result from relief of steric crowding due to the presence of four ring methyl substituents. Proper evaluation of the latter point will come as the structures of additional cyclic selenuranes are established.

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Registry No. **3**, 68351-64-4; **4**, 6069-58-5.

Supplementary Material Available: Compilations of anisotropic thermal parameters for $(C_6H_{12}O_2)_2Se$ (**3**) and $(C_6H_{12}O_2)_2Te$ (**4**) (Tables A and B, respectively), bond lengths and angles involving hydrogen atoms for both **3** and **4** (Table C), and observed and calculated structure factor amplitudes for **3** and **4** (12 pages). Ordering information is given on any current masthead page.

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