

1,4-oxatellurane moiety in  $C_4H_8OTeI_2$  are given in Table IV. Although the six-membered ring is in the chair form, the heteroatoms produce marked changes from cyclohexane geometry, as can be noted in Figures 3 and 4. A least-squares plane fitted to the carbon atoms is within 0.004 Å of each of the four atoms, a distance much less than the esd of the carbon positions, 0.011 Å. This least-squares plane makes dihedral angles of 27.5° with the C(1)-Te-C(4) plane and 70° with the C(2)-O-C(3) plane. The Te and O atoms are displaced 0.682 and -0.725 Å, respectively, from the carbon atom plane.

Because of the effects of secondary bonding on the lengths of the axial Te-I bonds, it is not very meaningful to compare the present observed values of 2.886 (1) and 2.938 (1) Å with those observed in previous studies of  $R_2TeI_2$  compounds. However, the present values do fall in the range of values 2.851 (2)-2.985 (2) Å observed in the earlier studies.<sup>5,6,8</sup> While  $\beta$ -dimethyltellurium diiodide has the formal composition  $Me_2TeI_2$ , its structure<sup>21</sup> is best described by the ionic formulation  $[Me_3Te]^+[MeTeI_4]^-$ . In this compound the Te-I bond distances range from 2.840 to 2.984 Å, close to the range of values observed in the nonionic  $R_2TeI_2$  compounds.

It is interesting that the crystal structure of  $C_4H_8OTeI_2$  differs so greatly from that of  $C_4H_8STeI_2$ <sup>6</sup> considering the

(21) F. Einstein, J. Trotter, and C. Williston, *J. Chem. Soc. A*, 2018 (1967).

fact that neither the oxygen atom nor the sulfur atom is involved in any intermolecular bonding or contacts less than the sums of the corresponding van der Waals radii. Apparently the effects of these atoms on the structures are transmitted through tellurium. A similar effect has been noted in the dissociation of  $C_4H_8OSeI_2$  and  $C_4H_8SSeI_2$  into the selenane and molecular iodine in dilute carbon tetrachloride solution.<sup>22</sup> The former compound has a dissociation constant 44% greater than that of the latter compound at 25°.

**Acknowledgments.** Support of part of this research by the National Science Foundation is gratefully acknowledged. The authors also thank the computing centers of the University of California at Davis and Los Angeles for use of their facilities and the Campus Computing Network at UCLA for a computing grant.

**Registry No.**  $C_4H_8OTeI_2$ , 41178-34-1.

**Supplementary Material Available.** A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 20× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D. C. 20036. Remit check or money order for \$3.00 for photocopy or \$2.00 for microfiche, referring to code number INORG-73-2665.

(22) J. D. McCullough and I. C. Zimmermann, *J. Phys. Chem.*, **65**, 888 (1961).

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## Crystal and Molecular Structure of Phenoxatellurin 10,10-Diiodide, $C_{12}H_8OTeI_2$ <sup>1</sup>

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Received April 17, 1973

The structure of phenoxatellurin 10,10-diiodide,  $C_{12}H_8OTeI_2$ , has been determined by use of three-dimensional X-ray data collected on an automated diffractometer with monochromatized Mo K $\alpha$  radiation. The space group is  $P2_1/n$  with four molecules in the unit cell for which  $a = 7.991$  (1) Å,  $b = 10.598$  (2) Å,  $c = 17.138$  (3) Å, and  $\beta = 103.69$  (1)°. The measured and calculated densities are 2.58 (2) and 2.589 (1) g cm<sup>-3</sup>, respectively. The structure was solved by the heavy-atom method and refined by full-matrix least-squares procedures to a conventional  $R$  index of 4.3%. The phenoxatellurin moiety consists of planar halves with a fold angle of 163.9 (4)° along the line of the shared Te and O atoms. Tellurium forms bonds with carbon atoms at 2.09 (2) and 2.11 (2) Å with a bond angle of 91.5 (6)°. Approximately perpendicular to the C-Te-C plane, Te forms axial bonds with I atoms at 2.945 (2) and 2.941 (2) Å. The I-Te-I angle is 176.44 (6)°. Tellurium also forms weak bonds with iodine atoms in each of two neighboring molecules with Te···I distances of 3.739 and 3.788 Å. The C-Te···I bond angles are 170.9 (6) and 171.0 (5)° so that the configuration about Te is octahedral. The intermolecular Te···I bonds link molecules into infinite chains along the  $a$  axis. The observed C-C bond distances range from 1.34 (3) to 1.46 (3) Å with an average of 1.39 Å. The C-O distances are 1.34 (2) and 1.37 (2) Å with a C-O-C bond angle of 125 (1)°.

### Introduction

Structural studies of di-*p*-chlorophenyltellurium diiodide,<sup>2</sup> 1-thia-4-telluracyclohexane 4,4-diiodide,<sup>3</sup> and 1-oxa-4-telluracyclohexane 4,4-diiodide<sup>4</sup> have shown that these compounds have extensive intermolecular bonding systems involving the

heavy atoms. Crystals of the first two compounds are dark violet and the second shows a definite metallic luster. The intermolecular bonding in these crystals consists mainly of nearly linear chains of the type ···I-Te-I···I-Te-I··· with secondary I···I bonds. In contrast, the third (or oxa) compound is bright red and the intermolecular bonds have been found to consist of Te···I interactions only. Interestingly, solutions of all molecular<sup>5</sup>  $R_2TeI_2$  compounds in organic solvents are yellow to orange. This suggests that the color of

(1) Presented, in part, as Paper No. K-9 at the National Meeting of the American Crystallographic Association, Albuquerque, N. M., April 1972.

(2) G. Y. Chao and J. D. McCullough, *Acta Crystallogr.*, **15**, 887 (1962).

(3) C. Knobler, J. D. McCullough, and H. Hope, *Inorg. Chem.*, **9**, 797 (1970).

(4) H. Hope, C. Knobler, and J. D. McCullough, *Inorg. Chem.*, **12**, 2665 (1973).

(5) Some modifications of alkyl  $R_2TeI_2$  compounds are ionic. For example, crystals of  $\beta$ -( $CH_3$ )<sub>2</sub>TeI<sub>2</sub> have been shown to consist of ( $CH_3$ )<sub>3</sub>Te<sup>+</sup> and  $CH_3TeI_4^-$  ions: F. Einstein, J. Trotter, and C. Williston, *J. Chem. Soc. A*, 2018 (1967).

unassociated  $R_2TeI_2$  molecules is yellow and that varying degrees and kinds of association in the solid state cause shifts in color to orange, red, or dark violet. The information available to date indicates that red crystals result when only  $Te \cdots I$  intermolecular bonding is involved and that the violet color and metallic luster of some  $R_2TeI_2$  compounds are the result of  $I \cdots I$  intermolecular bonds. An opportunity to test this implied relationship between the type of secondary (intermolecular) bonding and crystal color presented itself in the brilliant red crystals of phenoxatellurin diiodide. This was one reason for undertaking the present study; another was the determination of the structure of the interesting phenoxatellurin portion of the molecule.

### Experimental Section

Phenoxatellurin was prepared and purified by the method of Drew.<sup>6</sup> The diiodide was prepared by mixing solutions of phenoxatellurin and iodine in ethylene chloride in a nearly 1:1 molar ratio. On slow evaporation of the solvent, brilliant red crystals of the diiodide were formed. Nearly all of these were relatively thin plates on (001) which Drew described as "red spangles." A lengthy search of several batches under the microscope was required to find three specimens suitable for the X-ray study. The one finally chosen for the intensity measurements was approximately spherical in shape with dimensions  $0.087 \times 0.087 \times 0.067$  mm along  $a$ ,  $b$ , and  $c^*$ , respectively. A preliminary examination of the three specimens by means of Weissenberg and precession photographs showed monoclinic symmetry. The best specimen was mounted with  $a^*$  approximately along the  $\phi$  axis of a four-circle computer-controlled diffractometer (Syntex PI autodiffractometer) equipped with a scintillation counter and a graphite monochromator. By use of the automatic centering and indexing routines, a tentative unit cell was chosen which was used for the rapid collection (scan rate of  $24^\circ \text{ min}^{-1}$ ) of a shell of data in the  $2\theta$  range from  $20$  to  $25^\circ$ . Fifteen of the strongest reflections in this shell were selected and the automatic centering and indexing procedure was repeated. On the basis of these improved cell parameters, the intensity data were collected. Mo  $K\alpha$  radiation was used with scan rates which varied stepwise from  $24^\circ \text{ min}^{-1}$  in  $2\theta$  for the strongest reflections down to  $3^\circ \text{ min}^{-1}$  for the weakest. The scan range was from  $1.25^\circ$  below the  $K\alpha_1$  peak to  $1.25^\circ$  above the  $K\alpha_2$  peak. Background counts were taken for half the scan time at each end of the scan range. The pulse height analyzer was set at an 85% window for Mo  $K\alpha$  radiation.

The intensities of three reference reflections (400,  $13\bar{7}$ , and 244) were measured after every 48 intensity measurements. The variations in the measured values of these standards were random; 95% were within 4.0% of the respective mean values and no variation from mean was greater than 5.2%. In all, 2276 reflections were measured. These included 128 space group absences, 147 duplicate  $0kl$ ,  $0k\bar{l}$  pairs, and 144 periodic checks of the standard reflections. The remaining 1857 unique reflections represent all of those accessible within the range  $2\theta \geq 45^\circ$  for Mo  $K\alpha$  radiation, or all for which  $(\sin \theta)/\lambda \geq 0.539 \text{ \AA}^{-1}$ . Among these, 777 reflections with  $I \geq 3\sigma(I)$  were considered unobserved and were omitted from the refinement. The number of unique, observed reflections was thus 1080. An analysis of the  $|F_o|$  values of the 92 observed pairs of equivalent  $0kl$  reflections showed an average deviation of 2.2% from their respective mean values. The maximum deviation was less than  $1.5\sigma$  and 95% were within  $\sigma$ .

After collecting the intensity data, 15 of the stronger reflections in the  $2\theta$  range from  $30$  to  $45^\circ$  were recentered and the angular coordinates were processed by LATPAR<sup>7</sup> to obtain a more accurate set of lattice parameters. The resulting values at  $22^\circ$  are  $a = 7.991$  (1)  $\text{\AA}$ ,  $b = 10.598$  (2)  $\text{\AA}$ ,  $c = 17.138$  (3)  $\text{\AA}$ , and  $\beta = 103.69$  (1) $^\circ$  based on Mo  $K\alpha$ ,  $\lambda$  0.71069  $\text{\AA}$ . The measured density of 2.58 (2)  $\text{g cm}^{-3}$  compares favorably with the calculated value of 2.589 (1)  $\text{g cm}^{-3}$  for  $Z = 4$ . With the above unit cell, the systematic absences are  $0k0$  with  $k$  odd and  $h0l$  with  $h + l$  odd. The space group is thus indicated to be  $P2_1/n$ , which is an alternate setting of  $P2_1/c$  ( $C_{2h}^5$ , No. 14).<sup>8</sup> The

(6) H. D. K. Drew, *J. Chem. Soc.*, 223 (1926).

(7) The computer programs used included LATPAR by R. A. Sparks and M. R. Murphy for the least-squares refinement of lattice parameters from diffractometer angles and P1BAR by J. D. Bell and M. R. Murphy for reduction of data from the Syntex PI diffractometer. Other programs used were listed by C. Knobler and J. D. McCullough, *Inorg. Chem.*, 11, 3026 (1972). The index,  $R$ , was defined as  $\sum |F_o| - |F_c| / \sum |F_o|$ .

(8) "International Tables for X-Ray Crystallography," Vol. I, Kynoch Press, Birmingham, England, 1952, p 99.

Table I. Atomic Positional Parameters in  $C_{12}H_8OTeI_2$

Atom	$x$	$y$	$z$
Te	0.2530 (2) <sup>a</sup>	0.6396 (1)	0.5123 (1)
I(1)	0.5061 (2)	0.6557 (1)	0.4126 (1)
I(2)	0.0139 (2)	0.6115 (1)	0.6178 (1)
O	0.3300 (20)	0.9516 (12)	0.5165 (9)
C(1)	0.4135 (26)	0.7566 (15)	0.5980 (11)
C(2)	0.5105 (29)	0.7083 (20)	0.6659 (14)
C(3)	0.6235 (29)	0.7770 (19)	0.7221 (13)
C(4)	0.6269 (31)	0.9067 (22)	0.7106 (14)
C(5)	0.5301 (30)	0.9611 (20)	0.6427 (14)
C(6)	0.4148 (27)	0.8843 (18)	0.5826 (12)
C(7)	0.1844 (25)	0.9163 (18)	0.4652 (11)
C(8)	0.0969 (30)	1.0154 (19)	0.4183 (15)
C(9)	-0.0516 (28)	0.9935 (19)	0.3630 (13)
C(10)	-0.1235 (27)	0.8715 (22)	0.3493 (12)
C(11)	-0.0298 (36)	0.7752 (19)	0.3988 (18)
C(12)	0.1228 (25)	0.7961 (17)	0.4523 (11)
H(2) <sup>b</sup>	0.509 (23)	0.626 (16)	0.673 (10)
H(3)	0.671 (22)	0.730 (15)	0.786 (11)
H(4)	0.707 (23)	0.958 (17)	0.765 (11)
H(5)	0.561 (21)	1.063 (16)	0.642 (9)
H(8)	0.143 (24)	1.095 (18)	0.425 (10)
H(9)	-0.119 (21)	1.076 (15)	0.303 (9)
H(10)	-0.231 (23)	0.836 (16)	0.309 (11)
H(11)	-0.039 (31)	0.725 (18)	0.380 (13)

<sup>a</sup> The numbers given in parentheses here and in succeeding tables are the estimated standard deviations in the least significant digits.

<sup>b</sup> Hydrogen atoms are numbered to correspond to the carbon atoms to which they are bonded.

equivalent positions for this setting are  $A = (x, y, z)$ ,  $B = (1/2 + x, 1/2 - y, 1/2 + z)$ ,  $C = (1 - x, 1 - y, 1 - z)$ , and  $D = (1/2 - x, 1/2 - y, 1/2 - z)$ . With four molecules of  $C_{12}H_8OTeI_2$  in the unit cell, there is no required molecular symmetry.

The calculated value of  $\mu$  for Mo  $K\alpha$  radiation is  $66 \text{ cm}^{-1}$  and the above crystal dimensions indicate values of  $\mu r$  in the range 0.26-0.33. The latter value is based on the maximum "diameter" of the crystal between apices. If the crystal is assumed spherical, calculated values of the transmission factor range from 0.62 to 0.68 in the  $2\theta$  range involved. It is estimated that absorption effects would cause fluctuations of no more than  $\pm 5\%$  in  $I$  or  $\pm 2.5\%$  in  $|F_o|$ . A  $\psi$  scan of the  $h00$  reflections gave results consistent with this estimate and no corrections for the effects of absorption were made.

The data reduction and processing were carried out by use of P1BAR which makes use of the equations

$$I = N - (t_s/2t_b)(B_1 + B_2)$$

$$\sigma(I) = [N + (t_s/2t_b)^2(B_1 + B_2) + (0.04I)^2]^{1/2}$$

$$|F_o| = k[I/(Lp)]^{1/2}$$

$$\sigma(F_o) = (k/2)[\sigma(I)/I(Lp)]^{1/2}$$

In these equations  $N$  is the total scan count,  $B_1$  and  $B_2$  are the individual background counts,  $t_s$  is the scan time (sec), and  $t_b$  is the time (sec) each background is counted. In the present case  $t_s = 2t_b$  so that  $t_s/2t_b = 1$ .

### Determination and Refinement of the Structure

Trial positions for the tellurium and iodine atoms were obtained from a three-dimensional Patterson summation. Except for some initial difficulties caused by the apparent location of Te at  $x = 1/4$  and  $z = 1/2$  and the centering of the two iodine atoms about these positions, the solution of the structure was straightforward. The oxygen and carbon atoms were located by means of a difference Fourier and the positional and anisotropic thermal parameters of the sixteen non-hydrogen atoms were refined to a conventional  $R$  index of 0.048. A second difference Fourier served to locate all eight hydrogen atoms with maxima in the range from 0.4 to 0.6  $e \text{ \AA}^{-3}$ . Three more cycles of least-squares refinement were then computed in which the positional (but not the thermal) parameters of the hydrogen atoms were allowed to vary, along with the positional and anisotropic thermal parameters of the sixteen heavier atoms. In these cycles, the hydrogen atoms were assigned fixed isotropic thermal parameters of 5.0  $\text{\AA}^2$  and the  $R$  index decreased to its final value of 0.043. A set of structure factors<sup>9</sup> for all measured reflections was calculated on the basis of the final positional and thermal parameters given in Tables I and II.

Table II. Atomic Thermal Parameters in  $C_{12}H_8OTeI_2$ <sup>a,b</sup>

Atom	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
Te	116 (2)	053 (1)	031 (1)	007 (3)	003 (2)	001 (1)
I(1)	169 (3)	082 (1)	042 (1)	052 (3)	040 (2)	015 (1)
I(2)	193 (3)	099 (2)	042 (1)	-061 (4)	048 (2)	003 (2)
O	234 (36)	066 (14)	060 (8)	-055 (37)	003 (28)	035 (17)
C(1)	170 (45)	035 (17)	031 (9)	-063 (43)	023 (32)	-024 (19)
C(2)	175 (48)	081 (21)	039 (11)	006 (58)	-029 (35)	010 (26)
C(3)	204 (52)	083 (24)	031 (10)	-030 (56)	-010 (36)	-032 (24)
C(4)	223 (54)	109 (26)	033 (10)	-137 (64)	006 (37)	-056 (28)
C(5)	202 (51)	078 (23)	048 (12)	-084 (57)	108 (40)	-062 (26)
C(6)	166 (45)	081 (23)	034 (9)	-004 (51)	033 (31)	-023 (24)
C(7)	119 (39)	067 (20)	036 (9)	060 (49)	016 (30)	020 (22)
C(8)	176 (56)	058 (21)	055 (12)	031 (54)	062 (44)	048 (27)
C(9)	143 (49)	093 (23)	037 (10)	119 (55)	044 (36)	032 (25)
C(10)	124 (42)	134 (30)	034 (9)	039 (58)	-059 (31)	-010 (27)
C(11)	189 (60)	054 (24)	068 (16)	084 (62)	063 (49)	-010 (27)
C(12)	100 (40)	071 (21)	032 (9)	021 (45)	029 (30)	007 (21)

<sup>a</sup> All parameters have been multiplied by  $10^4$ . The anisotropic temperature factor expression is of the form  $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$ . The hydrogen atoms were assigned fixed isotropic thermal parameters of  $5.0 \text{ \AA}^2$ . <sup>b</sup> The root-mean-square amplitudes of vibration derived from the above thermal parameters are given in Table III.

(Table III gives rms amplitudes of vibration.) The atomic scattering factors used for iodine were the  $I^0$  values of Doyle and Turner<sup>10</sup> and their values for Sb<sup>9</sup> and I<sup>9</sup> were averaged to give a set of values for tellurium. The neutral-atom values given in Table 3.3.1A in Vol. III of ref 8 were used for oxygen and carbon, and the hydrogen factors were from Stewart, Davidson, and Simpson.<sup>11</sup> The real and imaginary anomalous dispersion corrections given for Mo  $K\alpha$  radiation in Table 3.3.2C of Vol. III of ref 8 were applied to the scattering factors for I and Te. An analysis of the values of  $|F_o| - |F_c|$  for the 777 unobserved reflections showed 82% to be less than  $\sigma(F_o)$ , 14% in the range from 1 to  $2\sigma(F_o)$ , and 4% in the range from 2 to  $3\sigma(F_o)$ . One reflection was beyond this range at  $4.1\sigma(F_o)$ . In the final least-squares cycle, the largest shift in a positional or thermal parameter for Te and I was  $0.10\sigma$ , for C and O  $0.20\sigma$ , and for the hydrogen positions  $0.4\sigma$ . The final "goodness of fit" defined as  $[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$  was 1.28. In this expression,  $N_o = 1080$ , the number of observed reflections, and  $N_v = 169$ , the number of variable parameters.

### Description and Discussion of the Structure

The phenoxatellurin diiodide molecule and the phenoxatellurin moiety are illustrated in Figures 1 and 2, respectively. The phenoxatellurin moiety consists of nearly planar halves which intersect along the Te-O line with a dihedral angle of  $163.9 (4)^\circ$ . Bond distances and angles for this portion of the molecule are given in Table IV and the average values for the chemically equivalent bonds and angles are shown in Figure 3. The benzene rings are each planar within the standard deviations ( $0.02 \text{ \AA}$ ) of the carbon atom positions but the tellurium and oxygen atoms show small displacements ( $0.04$  and  $0.16 \text{ \AA}$  for Te,  $0.00$  and  $0.06 \text{ \AA}$  for O) from the least-squares planes fit to the carbon atoms. The larger displacements, especially those for tellurium, are probably significant and may be due to the intermolecular bonding discussed below. A listing of atomic displacements from least-squares planes and some interplanar angles in the phenoxatellurin moiety are given in Table V.

A discussion of the structures of some molecules related to phenoxatellurin and a review of the relevant literature are given in a series of papers by Cordes and coworkers on the structures of some phenoxarsine derivatives.<sup>12-14</sup> The di-

(9) See paragraph at end of paper regarding supplementary material.

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(12) J. E. Stuckey, A. W. Cordes, L. B. Handy, R. W. Perry, and C. K. Fair, *Inorg. Chem.*, **11**, 1846 (1972).

(13) R. J. Holliday, R. W. Broach, L. B. Handy, A. W. Cordes, and L. Thomas, *Inorg. Chem.*, **11**, 1849 (1972).

(14) W. K. Grindstaff, A. W. Cordes, C. K. Fair, R. W. Perry, and L. B. Handy, *Inorg. Chem.*, **11**, 1852 (1972).

Table III. Root-Mean-Square Amplitudes of Vibration ( $\text{\AA}$ )

Atom	Min	Intermed	Max
Te	0.172	0.180	0.227
I(1)	0.194	0.237	0.252
I(2)	0.209	0.244	0.266
O	0.175	0.250	0.330
C(1)	0.113	0.218	0.239
C(2)	0.187	0.217	0.289
C(3)	0.165	0.239	0.277
C(4)	0.137	0.264	0.306
C(5)	0.160	0.200	0.312
C(6)	0.191	0.225	0.240
C(7)	0.152	0.221	0.235
C(8)	0.156	0.226	0.293
C(9)	0.151	0.217	0.277
C(10)	0.136	0.262	0.299
C(11)	0.143	0.257	0.310
C(12)	0.170	0.201	0.216

Table IV. Bond Distances and Angles within the Phenoxatellurin Moiety in  $C_{12}H_8OTeI_2$ 

(a) Distances, $\text{\AA}$			
Te-C(1)	2.11 (2)	Te-C(12)	2.09 (2)
O-C(6)	1.37 (2)	O-C(7)	1.34 (2)
C(1)-C(2)	1.34 (3)	C(12)-C(11)	1.36 (3)
C(2)-C(3)	1.36 (3)	C(11)-C(10)	1.42 (3)
C(3)-C(4)	1.39 (3)	C(10)-C(9)	1.41 (3)
C(4)-C(5)	1.36 (3)	C(9)-C(8)	1.35 (3)
C(5)-C(6)	1.46 (3)	C(8)-C(7)	1.40 (3)
C(6)-C(1)	1.38 (3)	C(7)-C(12)	1.37 (3)
C(2)-H(2)	0.9 (2)	C(11)-H(11)	0.6 (2)
C(3)-H(3)	1.2 (2)	C(10)-H(10)	1.0 (2)
C(4)-H(4)	1.1 (2)	C(9)-H(9)	1.4 (2)
C(5)-H(5)	1.1 (2)	C(8)-H(8)	0.9 (2)

(b) Average Bond Distances, $\text{\AA}$			
Te-C	2.10 (2)	C-C	1.39 (3)
O-C	1.36 (2)	C-H	1.0 (2)

(c) Bond Angles, Deg			
C(1)-Te-C(12)	91.5 (6)	C(6)-O-C(7)	125 (1)
Te-C(1)-C(2)	121 (1)	O-C(6)-C(5)	114 (2)
Te-C(12)-C(11)	118 (1)	O-C(7)-C(8)	114 (2)
Te-C(1)-C(6)	118 (1)	O-C(6)-C(1)	130 (2)
Te-C(12)-C(7)	123 (1)	O-C(7)-C(12)	126 (2)
C(1)-C(2)-C(3)	124 (2)	C(12)-C(11)-C(10)	123 (2)
C(2)-C(3)-C(4)	117 (2)	C(11)-C(10)-C(9)	115 (2)
C(3)-C(4)-C(5)	121 (2)	C(10)-C(9)-C(8)	122 (2)
C(4)-C(5)-C(6)	120 (2)	C(9)-C(8)-C(7)	121 (2)
C(5)-C(6)-C(1)	116 (2)	C(8)-C(7)-C(12)	120 (2)
C(6)-C(1)-C(2)	121 (2)	C(7)-C(12)-C(11)	120 (2)

<sup>a</sup> The standard deviations of the average values were obtained by use of the formula  $\sigma = [\sum_i^n (x_i - \bar{x})^2 / (n - 1)]^{1/2}$ .

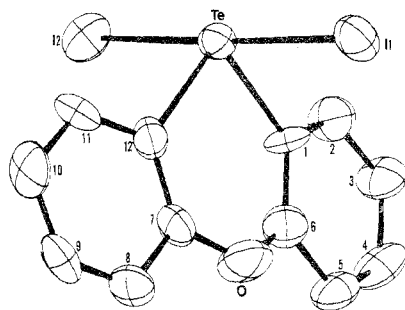
**Table V.** Results of Least-Squares Planes Calculations

(a) Distances of Atoms from Least-Squares Planes, $\text{\AA}$			
Atom	Plane A	Plane B	Plane C
Te	-0.04	-0.31	-0.15
O	-0.06	-0.21	0.00
C(1)	-0.01*	-0.01*	
C(2)	0.02*	0.18	
C(3)	-0.02*	0.32	
C(4)	0.02*	0.37	
C(5)	0.00*	0.19	
C(6)	0.00*	0.01*	
C(7)		-0.01*	0.02*
C(8)		0.11	0.00*
C(9)		0.29	0.00*
C(10)		0.35	-0.01*
C(11)		0.24	0.02*
C(12)		0.01*	-0.02*

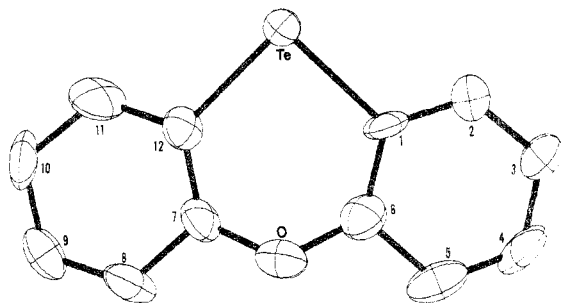
(b) Interplanar Angles, Deg

[Plane A]-[plane B]	171.9
[Plane B]-[plane C]	172.0
[Plane A]-[plane C]	163.9
[Plane B]-[C(1), Te, C(12)]	167.8
[Plane B]-[C(6), O, C(7)]	161.2
[Te, C(1), C(6), O]-[Te, C(12), C(7), O]	160.2

\* Atoms used to define the least-squares planes are indicated by asterisks.



**Figure 1.** ORTEP plot of the  $\text{C}_{12}\text{H}_8\text{OTeI}_2$  molecule. The thermal ellipsoids in this and the following figure are drawn at the 50% probability level. Hydrogen atoms are omitted from all figures for purposes of clarity. Carbon atoms are indicated by the numbers 1-12.



**Figure 2.** Plot of the phenoxatellurin moiety viewed normal to the plane of the four central carbon atoms.

hedral angle between the planes of the phenyl groups in these molecules is of particular interest. The angle of  $163.9^\circ$  observed in the phenoxatellurin moiety of the present study is in the range of values ( $156.3\text{--}178.5^\circ$ ) observed in the phenoxarsine derivatives and is also in the range ( $135\text{--}180^\circ$ ) observed in phenoxatellurin and three of its derivatives.<sup>15</sup> The dihedral angle observed in crystals of uncombined phen-

(15) E. A. Meyers, personal communication. The results of structural studies by Meyers and coworkers on phenoxatellurin, bis-(phenoxatellurin) dinitrate, phenoxatellurin dinitrate, and 10-acetonylphenoxatellurin nitrate were kindly provided to the present author prior to publication.

**Table VI.** Distances and Angles about Tellurium and Iodine Atoms in  $\text{C}_{12}\text{H}_8\text{OTeI}_2$ <sup>a</sup>

(a) Distances about Tellurium Atom, $\text{\AA}$			
Te-C(1)	2.11 (2)	Te-C(12)	2.09 (2)
Te-I(1)	2.945 (2)	Te-I(2)	2.941 (2)
Te $\cdots$ I(1)'	3.739 (2)	Te $\cdots$ I(2)''	3.788 (2)
(b) Angles about Tellurium Atom, Deg			
C(1)-Te-C(12)	91.5 (6)	I(1)-Te-I(2)	176.44 (6)
C(1)-Te-I(1)	89.0 (6)	C(12)-Te-I(1)	89.9 (6)
C(1)-Te-I(2)	90.4 (6)	C(12)-Te-I(2)	93.6 (6)
C(1)-Te $\cdots$ I(1)'	94.2 (5)	C(12)-Te $\cdots$ I(1)'	170.9 (6)
C(1)-Te $\cdots$ I(2)''	171.0 (5)	C(12)-Te $\cdots$ I(2)''	97.0 (5)
I(1)-Te $\cdots$ I(1)'	83.11 (5)	I(2)-Te $\cdots$ I(1)'	93.43 (5)
I(1)-Te $\cdots$ I(2)''	93.89 (5)	I(2)-Te $\cdots$ I(2)''	86.19 (5)
I(1)' $\cdots$ Te $\cdots$ I(2)''	77.71 (4)		
(c) Distances about Iodine Atoms, $\text{\AA}$			
I(1)-Te	2.945 (2)	I(2)-Te	2.941 (2)
I(1) $\cdots$ Te'	3.739 (2)	I(2) $\cdots$ Te''	3.788 (2)
(d) Angles about Iodine Atoms, Deg			
Te-I(1) $\cdots$ Te'	96.89 (5)	Te-I(2) $\cdots$ Te''	93.81 (5)

<sup>a</sup> Definition of symbols: unprimed atoms are in the  $\text{C}_{12}\text{H}_8\text{OTeI}_2$  molecule in symmetry position A of the origin cell, *i.e.* (A; 0, 0, 0); atoms with a single prime are in molecule (C; 0, 0, 0) and those with a double prime are in molecule (C; 1, 0, 0). The symmetry positions A, B, C, and D for the space group  $P2_1/n$  are given in the Experimental Section. Secondary (intermolecular) bonds are designated thus Te $\cdots$ I(1)'.

oxatellurin is  $145^\circ$ . The smaller dihedral angle in phenoxatellurin (compared to that in the diiodide) causes the bond angles at the heteroatoms in the inner ring to be smaller. Thus, the observed values in phenoxatellurin are C-Te-C =  $89.4 (3)^\circ$  and C-O-C =  $121.2 (5)^\circ$  compared to  $91.5 (6)$  and  $125 (1)^\circ$  in the diiodide. This effect in the phenoxarsine derivatives has been discussed by Cordes and coworkers.<sup>15</sup>

The twelve C-C distances in the benzene rings in the present study range from 1.34 to 1.46  $\text{\AA}$  with an average of 1.39  $\text{\AA}$  and the eight C-H distances range from 0.6 to 1.4  $\text{\AA}$  with an average of 1.0  $\text{\AA}$ . While these ranges appear large, they are reasonable in view of the  $\sigma$  values of 0.03 and 0.2  $\text{\AA}$  which apply to the C-C and C-H bond distances, respectively. The observed C-O distances of 1.34 and 1.37  $\text{\AA}$  average to 1.36  $\text{\AA}$  which is considered normal for O-C(aromatic) bonds. The two Te-C bonds (2.09 and 2.11  $\text{\AA}$ ) give an average of 2.10  $\text{\AA}$  which corresponds exactly to the sum of the radii for Te (1.37  $\text{\AA}$ ) and  $\text{sp}^2$  C (0.73  $\text{\AA}$ ).

While not required crystallographically, the phenoxatellurin diiodide molecule approximates mirror symmetry through the I, Te, and O atoms. The magnitude of the deviations from this symmetry may be derived from data given in Tables IV-VI. The bonding about tellurium in the free phenoxatellurin diiodide molecule might be considered to be trigonal bipyramidal with two carbon atoms and an unshared pair of electrons in the equatorial positions. In the crystalline solid, however, the configuration about Te is probably best described as octahedral. There are the two Te-C bonds mentioned earlier which make a C-Te-C bond angle of  $91.5^\circ$ . Approximately perpendicular to the C-Te-C plane, Te makes two axial bonds with iodine at distances of 2.945 and 2.941  $\text{\AA}$ . The I-Te-I bond angle is  $176.44^\circ$ . The octahedron about tellurium is completed by formation of secondary bonds from Te to an iodine atom in each of two neighboring molecules. These distances are 3.739 and 3.788  $\text{\AA}$ . For reference, the sum of the normal, single, covalent bond radii for Te and I is 2.70  $\text{\AA}$  and the sum of the van der Waals radii is 4.35  $\text{\AA}$ .<sup>16</sup> The C-Te $\cdots$ I angles made with the carbon

(16) L. Pauling, "The Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960, pp 224, 260.

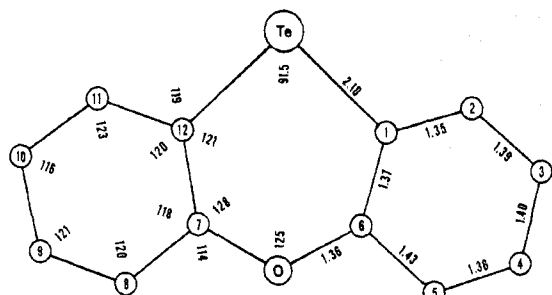
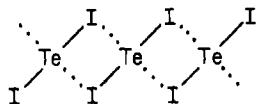


Figure 3. Drawing of the phenoxatellurin moiety showing average values of the chemically equivalent bonds and angles.

atoms opposite these loosely bonded iodine atoms are  $170.9$  and  $171.0^\circ$ . The remaining eleven bond angles about tellurium range from  $77.7$  to  $97.0^\circ$ , with nine of them within  $5^\circ$  of the ideal  $90^\circ$  value. A complete listing of the bond distances and angles about tellurium and iodine is given in Table VI.

The secondary  $\text{Te} \cdots \text{I}$  bonds link the molecules into infinite chains of the general type



These chains are parallel to the crystallographic  $a$  axis and the axial  $\text{I}-\text{Te}-\text{I}$  bonds are approximately perpendicular to the direction of the  $b$  axis. The light atoms have been omitted from the diagram for clarity. Alternate  $\text{I}-\text{Te}-\text{I}$  units are at different  $y$  levels so that the  $\text{I} \cdots \text{Te} \cdots \text{I}$  bond angles are near  $90^\circ$ . This type of intermolecular bonding has not been observed previously in  $\text{R}_2\text{TeI}_2$  compounds. An ORTEP plot of two chain segments as viewed down  $c^*$  is shown in Figure 4 and a plot of the coordination about tellurium is reproduced in Figure 5. The absence of  $\text{I} \cdots \text{I}$  intermolecular bonds in the red crystals of phenoxatellurin diiodide gives additional support to the idea that such bonds are responsible for the violet color observed in some  $\text{R}_2\text{TeI}_2$  compounds.

The axial  $\text{Te}-\text{I}$  bond lengths of  $2.945$  and  $2.941 \text{ \AA}$  are, as expected, longer than the sum of the normal, single, covalent bond radii ( $2.70 \text{ \AA}$ ) and are near the upper end of the range of values ( $2.851$ – $2.985 \text{ \AA}$ ) observed in previous studies.<sup>2-4</sup> These bonds correspond approximately to a bond order of  $1/2$  and the bond lengths in the various  $\text{R}_2\text{TeI}_2$  compounds are very sensitive to the strength of the secondary bonds formed by the iodine atoms involved. In the present compound, the longer axial  $\text{Te}-\text{I}$  bonds are probably the result of the shorter secondary bonds of  $3.739$  and  $3.788 \text{ \AA}$ , which are at the lower end of the range of values ( $3.739$ – $4.30 \text{ \AA}$ ) observed for  $\text{Te} \cdots \text{I}$  bonds.

The four nonbonded intramolecular separations between the iodine atoms and the carbon atoms bonded to tellurium are all in the range  $3.6$ – $3.7 \text{ \AA}$ , almost identical with the situation in 1-oxa-4-telluracyclohexane 4,4-diiodide.<sup>4</sup> Except for the  $\text{Te} \cdots \text{I}$  intermolecular bonds discussed above, there are no intermolecular contacts significantly less than the sums of the corresponding van der Waals radii.

**Acknowledgments.** The author is indebted to members of the UCLA Chemistry Department X-ray crystallography

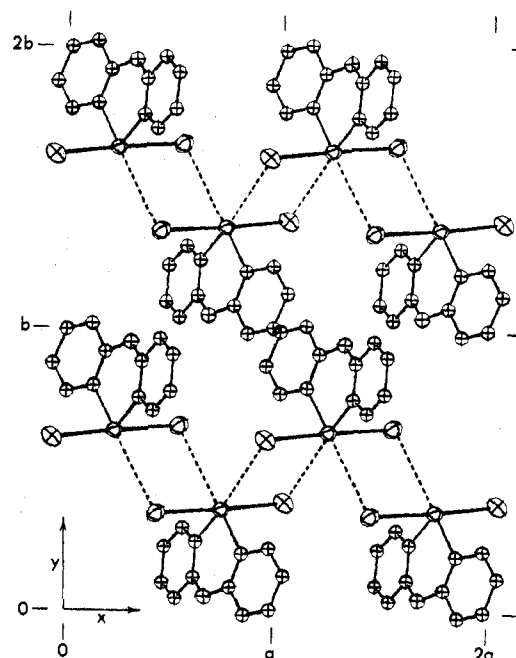


Figure 4. View down  $c^*$  showing segments of two of the chains formed by the  $\text{Te} \cdots \text{I}$  intermolecular bonds. The carbon atoms are shown with artificial, small isotropic thermal parameters.

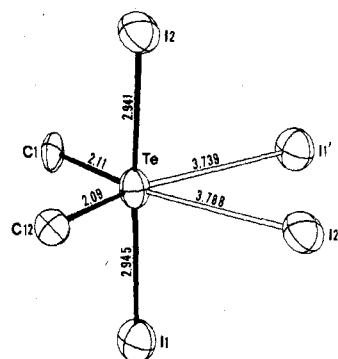


Figure 5. View of the coordination about tellurium. A complete listing of the angles involved is given in Table VI.

group and especially to Dr. J. D. Bell, Dr. G. W. Frank, Dr. Emily Maverick, and Mr. M. R. Murphy for help and advice on the use of the Syntex diffractometer. The financial assistance of the National Science Foundation in supporting part of this research through Grant GP-9428 and through Grant GP-28248 for purchase of the Syntex diffractometer is gratefully acknowledged. The author also thanks the UCLA Campus Computing Network for a grant of computing time and for use of computing equipment.

Registry No.  $\text{C}_{12}\text{H}_8\text{OTeI}_2$ , 31419-78-0.

**Supplementary Material Available.** A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche ( $105 \times 148 \text{ mm}$ ,  $20\times$  reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D. C. 20036. Remit check or money order for  $\$3.00$  for photocopy or  $\$2.00$  for microfiche, referring to code number INORG-73-2669.