Syntheses and Crystal Structures of Ethylenethiourea(iodo)-phenyltellurium(II) and Ethyleneselenourea(iodo)phenyltellurium(II)

OLAV VIKANE

Department of Chemistry, University of Bergen, N-5014 Bergen-Univ., Norway

The phenyltellurium(II) complexes, C_6H_5 Te-(etu)I, I, and C_6H_5 Te(esu)I, II, where etu = ethylenethiourea and esu = ethyleneselenourea, have been prepared from the corresponding bromo complexes through ligand exchange with sodium iodide in methanol, and the crystal structures have been determined by three-dimensional X-ray methods.

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The crystals of I and II are isomorphous, space group $P2_1/c$ with Z=4. The unit cell dimensions are: a=8.1441(14) Å, b=11.3750(12) Å, c=13.9243(16) Å, $\beta=105.181(9)$ ° for I, and a=8.2209(17) Å, b=11.3305(13) Å, c=14.1563

(19) Å, $\beta = 104.816(12)$ ° for II.

In the structures, each tellurium atom is three-coordinated, being bonded to one phenyl carbon atom and, in directions nearly perpendicular to the Te-C bond, to one iodine atom and one ethylenethiourea sulfur atom in I, and to one iodine atom and one ethyleneselenourea selenium atom in II. The three-centre systems S-Te-I and Se-Te-I are nearly linear, and the Te-C bond nearly bisects the angle of the three-centre system. The bond lengths and angles involving the tellurium atom are: Te-I=3.0033(12) Å, Te-S=2.614 (2) Å, Te-C=2.124(6) Å, \angle S-Te-I=175.88 (4)°, \angle S-Te-C=88.57(17)°, \angle I-Te-C=88.24(17)° in I, and Te-I=3.0951(14) Å, Te-Se=2.6791(18) Å, Te-C=2.112(7) Å, \angle Se-Te-I= 177.31(2)°, \angle Se-Te-C=89.92 (17)°, \angle I-Te-C=88.15(17)° in II.

Structures of three-coordinated tellurium(II) compounds reported recently include those of $C_6H_5Te(etu)Br$, $C_6H_5Te(etu)Cl$, $C_6H_5Te(esu)Br$, and the two anions $[C_6H_5Te(SCN)_2]^-$ and $[C_6H_5Te(SCN)_2]^{-3}$.

The first complexes of the type ArTe(L)X, where L is thiourea and X is chlorine or bromine, were prepared by Foss and Hauge in 1959,

and the crystal structures of $C_6H_5Te(tu)Cl$, $C_6H_5Te(tu)Br$, and $C_6H_5Te(tu)_2Cl$ have been determined.^{5–8} The tendency of tellurium(II) in its complexes is toward square-planar four-coordination, but when the phenyl group is one of the ligands, the tellurium(II) atom becomes three-coordinated, due to the pronounced *trans* bond-lengthening effect of the phenyl group.

The tellurium(II) complexes may be regarded as models for transition states in nucleophilic displacements at divalent tellurium.^{7,9} Bond lengths and angles in the three-coordinated as well as the four-coordinated tellurium(II) complexes indicate linear transition states. Furthermore, in the linear transition state the in- and outgoing groups are bonded, at 180°, through a single p orbital of the central tellurium atom.^{7,9}

EXPERIMENTAL

Ethylenethiourea(iodo)phenyltellurium(II), $C_6H_5Te(etu)I$, was prepared using 1 mmol (0.386 g) of bromo(ethylenethiourea)phenyltellurium(II), $C_6H_5Te(etu)Br,^{10}$ dissolved in 10 ml of warm methanol. To the solution was added 2 mmol (0.3 g) of sodium iodide dissolved in 5 ml of warm methanol. During the addition a colour change from orange red to deep red was observed. The solution was filtered while hot, and placed at room temperature for 5 h. Yield: 0.41 g (94 %). M.p. 116-118 °C (dec.). (Found: C 24.93; H 2.57; N 6.39; S 7.36; I 29.21. Calc. for $C_9H_{11}N_2STeI$: C 24.89; H 2.54; N 6.46; S 7.38; I 29.26).

Ethyleneselenourea(iodo)phenyltellurium(II), $C_6H_5Te(esu)I$, was prepared in the same way

as the ethylenethiourea analogue, using 1 mmol (0.43 g) of bromo(ethyleneselenourea)phenyltellurium(II), C_6H_5 Te(esu)Br, "dissolved in 10 ml of warm methanol, and adding 2 mmol (0.3 g) of sodium iodide dissolved in 5 ml of warm methanol. During the addition the colour of the solution changed from orange red to deep red. The solution was filtered while hot, and placed at room temperature for 3 h. Yield: 0.46 g (95 %). M.p. 137–139 °C (dec.). (Found: C 22.51; H 2.32; N 5.79; I 26.42. Calc. for $C_9H_{11}N_2$ SeTeI: C 22.47; H 2.29; N 5.83; I 26.40).

Space groups were determined from singlecrystal oscillation and Weissenberg photo-

graphs.

Methods used for data collection and reductions, and computational procedures are as described previously.3,12 For ethylenethiourea-(iodo)phenyltellurium(II), I, the measurements were performed on a crystal with the following dimensions, given as distances from the point of intersection of the crystal faces $(01\overline{1})$, (100), and $(0\overline{1})$: to (011), 0.093 mm; to $(0\overline{1})$, 0.062 mm; to $(\overline{1}00)$, 0.156 mm. The crystal used for ethyleneselenourea(iodo)phenyltellurium(II), II, had the following dimensions, given as distances from the point of intersection of the crystal faces $(01\overline{1})$, (100), and $(0\overline{11})$; to (011), 0.108 mm; to $(0\overline{1}1)$, 0.061 mm; to (100), 0.266 mm. The scale factors, based on the reference reflections, varied within 7 % for I, and 9 % for II. The lower limit for observed reflections was set equal to two times the standard deviation in net intensity. 2346 of 3211 independent reflections within $\theta = 28^{\circ}$ were found to be stronger than the lower limit for I. The corresponding numbers for II are 2595 of 3278.

CRYSTAL DATA

The crystals of I and II are brown red, isomorphous, monoclinic prisms, extended along the a axis. Systematic absences are: h0l for l odd, 0k0 for k odd. The space group is $P2_1/c$ (No. 14).

The unit cell dimensions were determined as described elsewhere. The \$\theta\$-values (all about 20°) of 31 reflections for I, and 36 reflections for II, were measured as described by Maartmann-Moe. The unit cell dimensions are: \$a = 8.1441(14)\$ Å, \$b = 11.3750(12)\$ Å, \$c = 13.9243(16)\$ Å, \$\beta = 105.181(9)°, \$Z = 4\$, \$D_x = 2.32\$ g/cm³, \$D_m = 2.31\$ g/cm³, \$\mu_{(MoK\alpha)} = 51.1\$ cm\$^{-1}\$ for I, and: \$a = 8.2209(17)\$ Å, \$b = 11.3305(13)\$ Å, \$c = 14.1563(19)\$ Å, \$\beta = 104.816(12)°, \$Z = 4\$, \$D_x = 2.50\$ g/cm³, \$D_m = 2.49\$ g/cm³, \$\mu_{(MoK\alpha)} = 79.1\$ cm\$^{-1}\$ for II.

Table 1. Atomic coordinates in fractions of monoclinic cell edges for ethylenethiourea(iodo)-phenyltellurium(II). Origin at a centre of symmetry. Standard deviations from least squares are given in parentheses.

	\boldsymbol{x}	y	z
Te	0.12590(5)	0.54450(4)	0.38028(3)
1	0.22215(6)	0.79411(4)	0.43960(4)
\mathbf{s}	0.0653(2)	0.32341(15)	0.33286(13)
C(1)	0.3472(7)	0.5338(5)	0.3248(4)
C(2)	0.3587(8)	0.6067(6)	0.2482(4)
C(3)	0.5043(9)	0.5976(7)	0.2114(5)
C(4)	0.6304(9)	0.5165(7)	0.2493(5)
C(5)	0.6160(8)	0.4418(6)	0.3289(5)
C(6)	0.4733(7)	0.4508(6)	0.3664(5)
C(7)	0.1585(7)	0.2522(5)	0.4426(4)
C(8)	0.2454(11)	0.0929(6)	0.5478(6)
C(9)	0.2970(10)	0.2067(6)	0.6073(5)
N(1)	0.1597(7)	0.1346(5)	0.4472(4)
N(2)	0.2362(7)	0.2985(5)	0.5282(4)

STRUCTURE DETERMINATION

The structures were solved by Patterson and Fourier methods and refined to R-values of 0.037 for I and 0.046 for II. Altogether 127 parameters were refined in each structure. The three-dimensional Fourier difference maps, based on the data of the final refinements, showed no peaks higher than 0.7 e/Å³ in either of the two structures.

Table 2. Atomic coordinates in fractions of monoclinic cell edges for ethyleneselenourea-(iodo)phenyltellurium(II). Origin at a centre of symmetry. Standard deviations from least squares are given in parentheses.

	\boldsymbol{x}	y	z
Те	0.12505(6)	0.53971(4)	0.37574(4)
Ι	0.21685(7)	0.79818(5)	0.43782(4)
\mathbf{Se}	0.06070(10)	0.31321(7)	0.32560(5)
C(1)	0.3441(7)	0.5320(5)	$0.3252(\hat{5})^{'}$
C(2)	0.3621(9)	0.6071(6)	0.2501(5)
C(3)	0.5082(10)	0.5994(7)	0.2160(6)
C(4)	0.6331(9)	0.5168(7)	0.2559(6)
C(5)	0.6191(8)	0.4426(7)	0.3322(6)
C(6)	0.4772(9)	0.4503(6)	0.3694(5)
C(7)	0.1646(9)	0.2406(7)	0.4439(5)
C(8)	0.2580(14)	0.0899(8)	0.5555(7)
C(9)	0.3062(10)	0.2065(7)	0.6080(6)
N(1)	0.1728(9)	0.1241(5)	0.4555(5)
N(2)	0.2395(9)	0.2928(6)	0.5280(4)

Table 3. Anisotropic thermal parameters (Å²) in the form $\exp - [2\pi^2(h^2a^{-2}U_{11} + \cdots + 2hka^{-1}b^{-1}U_{12} + \cdots)]$, for ethylenethiourea(iodo)phenyltellurium(II). The values have been multiplied by 10². Standard deviations in parentheses.

	U_{11}	$oldsymbol{U_{22}}$	$oldsymbol{U_{33}}$	$oldsymbol{U_{12}}$	$oldsymbol{U_{23}}$	U_{13}
Te	32.2(2)	40.2(2)	47.3(3)	1.8(2)	2.3(2)	11.3(2)
1	63.0(3)	39.5(2)	56.1(3)	-1.3(2)	-6.1(2)	17.4(2)
S	45.2 (10)	44.5(10)	45.7(10)	-9.6(8)	-2.8(8)	7.4(8)
C(1)	30(3)	39(3)	41(3)	-6(3)	-6(3)	7(3)
C(2)	50(4)	51(4)	35(4)	– 7(3)	-1(3)	8(3)
C(3)	49(4)	66(5)	42(4)	$-1\dot{5}(4)$	-1(4)	1 4 (3)
C(4)	45(4)	66(5)	46(4)	-7(4)'	-11(4)	13(3)
C(5)	38(4)	52(4)	50(4)	-2(3)	-13(3)	9(3)
C(6)	34(3)	39(3)	51(4)	1(3)	-5(3)	5(3)
C(7)	30(3)	37(3)	40(4)	-4(3)	-2(3)	14(3)
C(8)	92(6)	43(4)	58(5)	7(4)	1(4)	8(5)
C(9)	7 5(5)	36(4)	51(4)	 7(4)	11(3)	1(4)
N(1)	54(4)	30(3)	65(4)	0(3)	-3(3)	18(3)
N(2)	50(3)	44 (3)	42 (3)	-7(3)	-1(3)	10(3)

Observed and calculated structure factors for the two structures are available from the author.

The atomic coordinates and thermal parameters are listed in Tables 1-4.

RESULTS

Bond lengths and angles in ethylenethiourea. (iodo)phenyltellurium(II), I, and ethyleneselenourea(iodo)phenyltellurium(II), II, based on the atomic coordinates in Tables 1 and 2,

are listed in Tables 5 and 6. The uncertainties in the unit cell dimensions are taken into account in the given standard deviations. Views of the structures of I and II, as seen normal to the plane through the coordination group, are shown in Figs. 1 and 2. Stereoscopic views of the content of the unit cell of I and II are shown in Figs. 3 and 4.

In each of the structures the tellurium atom is three-coordinated, being bonded to one phenyl carbon atom and, in directions nearly perpendicular to the Te-C bond, to one iodine

Table 4. Anisotropic thermal parameters (Å²) in the form $\exp - [2\pi^2(h^2a^{-2}U_{11} + \cdots + 2hka^{-1}b^{-1}U_{12} + \cdots)]$, for ethyleneselenourea(iodo)phenyltellurium(II). The values have been multiplied by 10³. Standard deviations in parentheses.

	U_{11}	${\pmb U}_{{f 22}}$	$oldsymbol{U_{33}}$	U_{12}	${m U}_{23}$	${U}_{13}$
Te	33.1(2)	36.7(3)	49.8(3)	1.1(2)	2.4(2)	13.1(2)
I	66.0(4)	37.5(3)	56.4(3)	-1.9(3)	-7.5(3)	18.9(3)
Se	36.9(4)	34.4(4)	40.2(4)	-5.6(4)	-2.3(3)	7.0(3)
C(1)	18(3)	25(3)	47(4)	8(3)	-5(3)	6(3)
C(2)	42(4)	49(4)	55(5)	-6(4)	-10(4)	3(4)
C(3)	4 5(5)	59(5)	59(5)	-6(4)	-12(4)	10(4)
C(4)	34(4)	57(5)	55(S)	-3(4)	-18(4)	9(4)
C(5)	29(4)	47(5)	56(5)	$2\dot{2}(3)$	-12(4)	7(4)
C(6)	40(4)	37(4)	50(4)	21(4)	$-5(\hat{4})'$	4 (3)
C(7)	4 0(4)	55(5)	36(4)	$-5(\hat{4})'$	-7(4)	21(3)
C(8)	117(9)	46(5)	56(6)	-3(6)	-1(5)	-3(6)
C(9)	52(5)	38(4)	54 (5)	-3(4)	0(4)	5(4)
N(1)	76(5)	32(3)	51(4)	2(4)	1(3)	15(4)
N(2)	58(4)	46(4)	38(3)	-10(3)	-4(3)	12(3)

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Table 5 Bond lengths (Å) and angles (°) in ethylenethiourea(iodo)phenyltellurium(II). Standard deviations are given in parentheses.

Te-I $Te-S$ $Te-C(1)$	3.0033(12) 2.614(2) 2.124(6)	175.88(4) 88.57(17) 88.24(17)
$\begin{array}{l} C(1) - C(2) \\ C(1) - C(6) \\ C(2) - C(3) \\ C(3) - C(4) \\ C(4) - C(5) \\ C(5) - C(6) \end{array}$	1.373(9) 1.403(8) 1.413(11) 1.378(10) 1.426(10) 1.397(10)	118.7(4) 118.9(4) 122.4(5) 118.1(5) 121.5(6) 119.4(6) 119.5(5) 119.0(5)
$\begin{array}{l} S-C(7) \\ C(7)-N(1) \\ C(7)-N(2) \\ N(1)-C(8) \\ N(2)-C(9) \\ C(8)-C(9) \end{array}$	1.720(6) 1.340(8) 1.305(7) 1.470(9) 1.505(8) 1.535(10)	102.6(2) 120.6(4) 128.1(4) 111.3(5) 111.3(5) 112.1(5) 103.7(5) 101.4(5)

atom and one ethylenethiourea sulfur atom in I, and to one iodine atom and one ethylene-selenourea selenium atom in II.

The coordination around the tellurium atom is nearly planar in both structures. The largest deviation from a least-squares plane through Te, S, I and C(1) in I is 0.043 Å, and the largest deviation from the corresponding plane through

Te, Se, I and C(1) in II is 0.029 Å. The least-squares plane through the coordination group passes 0.011 Å from C(4) in I and 0.032 Å from C(4) in II.

The three-centre systems S-Te-I and Se-Te-I are nearly linear. The S-Te-I bond angle in I is 175.88(4)°, and the Se-Te-I bond angle in II is 177.31(2)°. Similar devia-

Table 6. Bond lengths (Å) and angles (°) in ethyleneselenourea(iodo)phenyltellurium(II). Standard deviations are given in parentheses.

$egin{array}{l} { m Te-I} \\ { m Te-Se} \\ { m Te-C(1)} \end{array}$	3.0951(14) 2.6791(18) 2.112(7)	177.31(2) 89.92(17) 88.15(17)
C(1) - C(2) $C(1) - C(6)$ $C(2) - C(3)$ $C(3) - C(4)$ $C(4) - C(5)$ $C(5) - C(6)$	1.399(10) 1.448(9) 1.407(12) 1.397(11) 1.397(12) 1.399(11)	120.0(4) 119.9(5) 120.1(6) 119.2(6) 120.5(7) 121.2(7) 119.7(6) 119.2(6)
$\begin{array}{c} \mathrm{Se} - \mathrm{C}(7) \\ \mathrm{C}(7) - \mathrm{N}(1) \\ \mathrm{C}(7) - \mathrm{N}(2) \\ \mathrm{N}(1) - \mathrm{C}(8) \\ \mathrm{N}(2) - \mathrm{C}(9) \\ \mathrm{C}(8) - \mathrm{C}(9) \end{array}$	1.866(7) 1.329(10) 1.330(9) 1.462(11) 1.491(9) 1.518(11)	99.8(2) 123.0(5) 127.4(5) 109.5(6) 112.3(6) 112.6(6) 104.1(6) 101.5(6)

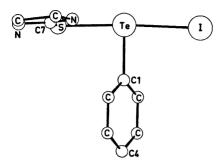


Fig. 1. Ethylenethiourea(iodo)phenyltellurium (II), as seen normal to the plane through the coordination group.

tions from linearity are also found in the crystals of chloro- and bromo(ethylenethiourea)-phenyltellurium(II),^{1,2} bromo(ethyleneselenourea)phenyltellurium(II),² and chloro- and bromo(phenyl)thioureatellurium(II),⁵⁻⁷ where the bond angle of the three-centre system varies from 171.9(5) to 177.22(3)°.

The Te-C bond nearly bisects the angle of the three-centre system in both structures. The S-Te-C bond angle is $88.57(17)^{\circ}$, and the Se-Te-C bond angle is $89.92(17)^{\circ}$. The I-Te-C bond angle is $88.24(17)^{\circ}$ in I and $88.15(17)^{\circ}$ in II.

The Te-S bond length, 2.614(2) Å in I, is about 0.092 Å longer than the mean Te-S bond length, 2.522 Å, found in the crystals of chloro- and bromo(ethylenethiourea)phenyltellurium(II),^{1,2} and about 0.204 Å longer than the sum of the single-bond radii.¹⁴ The Te-Se bond length, 2.6791(18) Å in II, is about 0.063 Å longer than the Te-Se bond length, 2.616 Å found in the crystals of bromo(ethyleneselenourea)phenyltellurium(II),² and about

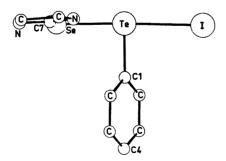


Fig. 2. Ethyleneselenourea(iodo)phenyltellurium(II), as seen normal to the plane through the coordination group.

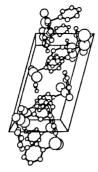
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0.139 Å longer than the sum of the single-bond radii¹⁴ Thus, it is seen that substitution of iodine for chlorine or bromine causes an increase in the length of the *trans*-positioned Te-S or Te-Se bond of 0.092 and 0.063 Å, respectively. Substitution of bromine for chlorine is known to cause no significant lengthening of the *trans*-positioned Te-ligand bond. 1,2,5-7 Thus, the iodine atom appears to have a far more pronounced *trans* bond-lengthening effect on a Te-S or a Te-Se bond than has chlorine or bromine. This indicates the following order of relative *trans* bond-lengthening effect of halogens on a Te-Se or a Te-Se bond: $I>Br \sim Cl$.

The Te-I bond length, 3.0033(12) Å in I, and 3.0951(14) Å in II, is 0.303 and 0.395 Å, respectively, longer than the sum of the single-bond radii. The 0.092 Å shorter Te-I bond length in I as compared with II, indicates a stronger Te-I bond when the *trans*-positioned ligand is ethylenethiourea than when it is ethyleneselenourea. Furthermore, the *trans*-bond-lengthening effect of iodine on a Te-S bond is more pronounced than the effect on a Te-Se bond.

As pointed out by Foss,7,9 the tellurium(II) complexes may be regarded as model substances for transition states in nucleophilic displacements on divalent tellurium. From several works on the preparation of tellurium(II) complexes, 4,6,15,16 it is known that chlorine, bromine, and iodine displace each other in the order mentioned. Nucleophilic reactivity may then relate to the ability of the reagent to engage the p orbital of the electrophilic centre in bonding, at the expense of the bond at 180°.9 This is in accord with the relative trans bond-lengthening order of bromine and iodine on a Te-S or a Te-Se bond indicated above. That bromine displaces chlorine is, however, not reflected in the trans bondlengthening effects of chlorine and bromine.

The Te-C bond lengths, 2.124(6) Å in I and 2.112(7) Å in II, are equal within the accuracies of the structure determinations. The same Te-C bond length has been found in the structures of chloro- and bromo(ethylenethiourea)phenyltellurium(II), bromo(ethyleneselenourea)phenyltellurium(II), the phenyldithiocyanato- and the phenyldiselenocyanato-tellurate(II) ions, chloro- and bromo(phenyl)



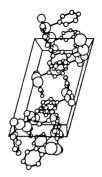


Fig. 3. A stereoscopic view of the cell packing in ethylenethiourea (iodo) phenyltellurium (II), as seen along the b crystal axis.

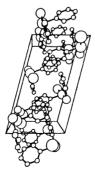
thioureatellurium(II),⁵⁻⁷ phenylbis(thiourea)-tellurium(II) chloride,⁶⁻⁸ and o-formylphenyl-tellurenyl bromide,¹⁷ indicating that the Te-C bond is a single covalent bond.³

The coordination around the tellurium atom may be regarded as based on a square-planar arrangement.^{6,7} In the structures of the isomorphous forms of chloro- and bromo(ethylene-thiourea)phenyltellurium(II),^{1,2} and in bromo-(ethylene-selenourea)phenyltellurium(II),² the fourth position of a square-planar coordination, trans to the phenyl group, is approached by a halogen atom of an adjacent molecule, over a symmetry centre. In the two present structures, as in the structure of the second form of bromo(ethylenethiourea)phenyltellurium(II),¹ this approach of a fourth ligand is missing.

The tellurium atom and the carbon atoms of the phenyltellurium group are nearly coplanar in both structures, the largest deviation from a least-squares plane being 0.019 Å in I and 0.035 Å in II. The angle between this plane and the least-squares plane through

the coordination group is 60.43° in I and 61.18° in II. In the structure of the C2/c form of bromo-(ethylenethiourea)phenyltellurium(II) ¹ the angle between these two least-squares planes is 64.6° .

The sulfur atom, the two nitrogen atoms and the three carbon atoms of the ethylenethiourea group in I are nearly co-planar. So are the selenium atom, the two nitrogen atoms and the three carbon atoms of the ethyleneselenourea group in II. The largest deviation from the least-squares plane is 0.019 Å in I and 0.009 Å in II. The angle between this plane and a least-squares plane through Te, S, I, and C(7) in I, or through Te, Se, I, and C(7) in II, is 5.37 and 4.28°, respectively. In the structure of the C2/c form of bromo(ethylenethiourea) phenyltellurium (II) 1 the atoms of the ethylenethiourea group lie nearly in the plane through Te, S, Br, and C(7). Thus, the ethylenethiourea group in I and the ethyleneselenourea group in II have the same orientation as the ethylenethiourea group in the C2/c



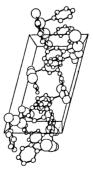


Fig. 4. A stereoscopic view of the cell packing in ethyleneselenourea(iodo)phenyltellurium(II), as seen along the b crystal axis.

form of bromo(ethylenethiourea)phenyltellurium(II). 1

The S-C bond length, 1.720(6) Å, and the Te-S-C bond angle, 102.6(2)°, in I are, within the error, equal to the S-C bond lengths and Te-S-C bond angles found in the structures of chloro- and bromo(ethylenethiourea)phenyltellurium(II).^{1,2}The corresponding Se-C bond length, 1.866(7) Å, and Te-Se-C bond angle, 99.8(2)°, in II are, within the error, equal to the Se-C bond length and Te-Se-C bond angle found in the structure of bromo(ethyleneselenourea)-phenyltellurium(II).²

CRYSTAL PACKING

Stereoscopic views of the content of the unit cell of I and II are shown in Figs. 3 and 4. In both structures there are no particular short non-bonding distances between the different molecules, Thus, the crystals of I and II consist of well separated ethylenethiourea(iodo)phenyltellurium(II) molecules in I, and ethyleneselenourea(iodo)phenyltellurium-(II) molecules in II.

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