

The Crystal Structure of Phenylbis(selenourea)tellurium(II) Chloride and Refinement of the Crystal Structure of Phenylbis(thiourea)tellurium(II) Chloride

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Phenylbis(selenourea)tellurium(II) chloride, $C_6H_5Te(su)_2Cl$, I, forms triclinic crystals with space group $P\bar{1}$, and phenylbis(thiourea)tellurium(II) chloride, $C_6H_5Te(tu)_2Cl$, II, forms orthorhombic crystals with space group $P2_12_12_1$. The unit cell dimensions for I are: $a = 11.2593(8)$ Å, $b = 12.1588(4)$ Å, $c = 5.9748(4)$ Å, $\alpha = 99.029(6)^\circ$, $\beta = 93.966(5)^\circ$, $\gamma = 62.507(3)^\circ$, and for II: $a = 11.9509(5)$ Å, $b = 20.7517(7)$ Å, $c = 5.7816(3)$ Å.

In I and II each tellurium atom is primarily three-coordinated, being bonded to one phenyl carbon atom and, in directions nearly perpendicular to the Te–C bond, to two selenourea selenium atoms in I, and to two thiourea sulfur atoms in II. The three-centre systems Se–Te–Se and S–Te–S are nearly linear, and the Te–C bond nearly bisects the angle of the three-centre system. The bond lengths and angles involving tellurium are: Te–Se = 2.7229(16) Å, 2.8895(17) Å; Te–C = 2.129(6) Å; Se–Te–Se = 173.09(4)°; Se–Te–C = 88.71(15)°, 86.65(16)° in I, and Te–S = 2.616(2) Å, 2.766(3) Å; Te–C = 2.102(7) Å; S–Te–S = 172.37(8)°; S–Te–C = 87.9(2)°, 84.6(2)° in II. In II, the fourth position of a square-planar arrangement around tellurium is approached by the chloride ion, which lies 3.578(2) Å from tellurium in a direction which makes an angle of 163.8(2)° with the direction of the Te–C bond.

The crystal structures of several three-coordinated complexes of divalent tellurium have been reported.^{1–6} The present paper reports the crystal structure determination of phenylbis(selenourea)tellurium(II) chloride, and the refinement of the crystal structure of phenylbis(thiourea)tellurium(II) chloride. The crystal structure of the latter com-

pound was reported by Foss and Marøy in 1966.¹ The refinement of the latter structure was undertaken in order to gain more accurate structural parameters. The syntheses and crystal data of the two present compounds are reported earlier.^{1,7,8}

EXPERIMENTAL

Methods used for data collections and reductions, and computational procedures are described elsewhere.^{3,9} For phenylbis(selenourea)tellurium(II) chloride, $C_6H_5Te(su)_2Cl$, I, the measurements were performed on a crystal with the following dimensions, given as distances from the point of intersection of the crystal faces (001), (010), and (100): to (00 $\bar{1}$), 0.134 mm; to (0 $\bar{1}$ 0), 0.059 mm; to (100), 0.074 mm; to (11 $\bar{1}$), 0.067 mm; to ($\bar{1}$ 11), 0.037 mm. For phenylbis(thiourea)tellurium(II) chloride, $C_6H_5Te(tu)_2Cl$, II, the crystal used had the following dimensions, given as distances from the point of intersection of the crystal faces (120), ($\bar{1}$ 10), and (001): to (00 $\bar{1}$), 0.253 mm; to ($\bar{1}$ 10), 0.061 mm; to (110), 0.036 mm. The intensities were corrected for absorption.

The net count of the reference reflections varied within 11% for I, and within 2.5% for II. 2641 of 4177 independent reflections within $\theta = 30^\circ$ were found to be stronger than the lower limit for I. The corresponding numbers for II are 1417 of 2428.

CRYSTAL DATA

The crystals of $C_6H_5Te(su)_2Cl$, I, are yellow, triclinic prisms with space group $P\bar{1}$ (No. 2). The crystals of $C_6H_5Te(tu)_2Cl$, II, are long orthorhombic prisms, extended along the c -axis. Systematic

Table 1. Positional and thermal parameters. The anisotropic temperature factor is $\exp[-2\pi^2(U_{11}h^2 + a^*2 + \dots + 2U_{12}hka^*b^* + \dots)]$. The isotropic temperature factor is $\exp[-8\pi^2U(\sin^2\theta/\lambda^2)]$. The values of the anisotropic temperature factors have been multiplied by 10^3 .

 $C_6H_5Te(su)_2Cl$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₂₃	<i>U</i> ₁₃
Te	0.37686(5)	0.45416(4)	0.74729(7)	47.8(3)	31.1(2)	34.1(2)	-15.5(2)	7.4(2)	7.8(2)
Se(1)	0.64560(7)	0.31850(6)	0.67456(11)	34.3(4)	40.7(4)	36.7(4)	-12.8(3)	15.5(3)	-3.3(3)
Se(2)	0.09158(7)	0.56967(6)	0.82625(11)	36.5(4)	30.6(4)	34.2(4)	-8.2(3)	10.0(3)	-2.3(3)
Cl	0.87985(18)	0.95967(16)	0.2812(3)	62.6(11)	46.4(9)	44.4(10)	-27.2(9)	17.8(8)	-13.5(8)
C(1)	0.3480(6)	0.3254(5)	0.4921(9)	36(3)	34(3)	34(3)	-12(3)	2(3)	-4(3)
C(2)	0.3858(7)	0.2045(6)	0.5353(12)	72(5)	33(4)	62(5)	-20(4)	9(3)	-19(4)
C(3)	0.3607(9)	0.1225(7)	0.3677(14)	85(6)	42(4)	79(6)	-26(4)	2(4)	-21(5)
C(4)	0.3052(7)	0.1607(7)	0.1573(12)	61(5)	55(5)	57(5)	-27(4)	-2(4)	-10(4)
C(5)	0.2677(7)	0.2800(7)	0.1220(11)	41(4)	60(4)	41(4)	-21(4)	1(3)	0(3)
C(6)	0.2879(6)	0.3664(6)	0.2890(9)	37(4)	50(4)	29(3)	-16(3)	5(3)	-3(3)
C(7)	0.6924(6)	0.2021(6)	0.8826(10)	42(4)	42(4)	32(3)	-18(3)	4(3)	-5(3)
C(8)	0.0332(6)	0.6952(6)	0.6351(10)	30(3)	36(3)	43(4)	-11(3)	9(3)	5(3)
N(1)	0.8088(6)	0.1030(6)	0.8555(11)	52(4)	62(4)	85(5)	3(3)	39(4)	9(4)
N(2)	0.6161(6)	0.2175(6)	1.0548(9)	61(4)	66(4)	42(3)	-23(3)	15(3)	8(3)
N(3)	-0.0088(6)	0.6762(5)	0.4298(9)	79(5)	45(3)	39(3)	-22(3)	10(3)	-15(3)
N(4)	0.0390(7)	0.7999(5)	0.7114(11)	93(5)	40(3)	73(4)	-34(4)	13(3)	-14(4)

 $C_6H_5Te(tu)_2Cl$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₂₃	<i>U</i> ₁₃
Te	0.42660(5)	0.11802(3)	0.36053(12)	43.5(3)	49.4(3)	45.9(3)	10.0(4)	7.3(4)	2.4(4)
S(1)	0.4084(2)	0.00884(11)	0.5836(4)	74.2(18)	42.8(13)	33.0(14)	2.2(14)	6.2(11)	5.7(14)
S(2)	0.47597(19)	0.23250(11)	0.1380(5)	48.9(13)	60.5(15)	50.4(14)	-12.4(12)	17.5(17)	-13.5(17)
Cl	0.14972(19)	0.18005(12)	0.4370(4)	48.3(14)	64.2(16)	35.7(14)	6.5(12)	5.7(12)	2.5(11)
C(1)	0.6010(6)	0.1053(4)	0.3675(18)	36(5)	35(5)	48(5)	2(4)	0(5)	3(5)
C(2)	0.6577(9)	0.0746(5)	0.1919(19)	49(6)	54(6)	57(7)	7(5)	0(6)	-2(6)
C(3)	0.7748(9)	0.0631(6)	0.206(3)	44(7)	70(8)	114(12)	13(6)	28(8)	21(8)
C(4)	0.8316(9)	0.0849(6)	0.398(3)	51(7)	67(8)	97(11)	8(6)	27(8)	-16(9)
C(5)	0.7768(9)	0.1160(7)	0.575(2)	63(7)	76(8)	76(9)	-11(8)	26(9)	-24(7)
C(6)	0.6599(9)	0.1282(5)	0.5650(17)	62(7)	56(7)	48(6)	-8(6)	15(6)	-5(5)
C(7)	0.4040(7)	-0.0498(4)	0.3739(17)	40(5)	52(5)	41(5)	-12(4)	-1(6)	-4(6)
C(8)	0.3778(8)	0.2392(4)	-0.0749(15)	59(6)	28(5)	32(5)	-1(4)	-2(4)	-2(5)
N(1)	0.3922(6)	-0.0367(4)	0.1489(15)	67(5)	74(5)	30(4)	-1(4)	9(5)	8(5)
N(2)	0.4150(9)	-0.1108(4)	0.4397(13)	144(8)	40(4)	53(5)	-19(7)	-2(4)	2(6)
N(3)	0.4085(7)	0.2618(3)	-0.2797(12)	71(6)	49(5)	43(5)	-3(5)	10(4)	-3(5)
N(4)	0.2705(6)	0.2234(5)	-0.0373(15)	33(5)	128(8)	60(6)	-26(5)	23(6)	-10(5)

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H(2)	0.611(7)	0.058(4)	0.075(18)	0.08(4)
H(3)	0.849(10)	0.041(5)	0.10(2)	0.15(5)
H(4)	0.938(8)	0.071(4)	0.441(18)	0.11(3)
H(5)	0.819(7)	0.131(5)	0.730(16)	0.07(3)
H(6)	0.587(10)	0.160(6)	0.74(2)	0.22(6)

absences are: $h00$ for h odd, $0k0$ for k odd, $00l$ for l odd. The space group is $P2_12_12_1$ (No. 19).

The unit cell dimensions were determined as described elsewhere.⁴ The θ -values (all about 20°) of 21 reflections for I, and 30 reflections for II were measured as described by Maartmann-Moe.¹⁰ The unit cell dimensions are: $a = 11.2593(8)$ Å, $b = 12.1588(4)$ Å, $c = 5.9748(4)$ Å, $\alpha = 99.029(6)^\circ$, $\beta = 93.966(5)^\circ$, $\gamma = 62.507(3)^\circ$. $Z = 2$, $D_x = 2.28$ g/cm³, $D_m = 2.30$ g/cm³, $\mu(\text{MoK}\alpha) = 78.54$ cm⁻¹ for I, and: $a = 11.9509(5)$ Å, $b = 20.7517(7)$ Å, $c = 5.7816(3)$ Å, $Z = 4$, $D_x = 1.81$ g/cm³, $D_m = 1.81$ g/cm³, $\mu(\text{MoK}\alpha) = 25.96$ cm⁻¹ for II.

STRUCTURE DETERMINATION

The structure of I was solved by Patterson and Fourier methods, and for II the coordinates given by Foss and Marøy¹ were used. Of the hydrogen atoms, only positions of the phenyl hydrogen atoms in II were located. The final full-matrix least-squares refinements, with weighting scheme, $w = 1/\sigma^2(F)$, resulted in the R values 0.037 for I and 0.038 for II. Altogether 145 parameters were refined for I and 165 parameters for II.

Observed and calculated structure factors for the two structures are available from the authors. The atomic coordinates and thermal parameters are listed in Table 1.

RESULTS

Bond lengths and angles in phenylbis(selenourea)-tellurium(II) chloride and phenylbis(thiourea)tellurium(II) chloride, based on the atomic coordinates in Table 1, are listed in Tables 2 and 3. The uncertainties in the unit cell dimensions are taken into account in the given standard deviations.

Views of the structure 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.

The structure of phenylbis(thiourea)tellurium(II) chloride has previously been described by Foss and Marøy.¹ In each of the two present 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 two selenourea selenium atoms in I, and to two thiourea sulfur atoms in II.

The coordination around the tellurium atom is nearly planar in each structure. The largest deviation from a least-squares plane through Te, Se(1), Se(2), and C(1) is 0.07 Å in I. The largest deviation from the corresponding least-squares plane through Te, S(1), S(2), and C(1) is 0.023 Å in II. The least-squares plane through the coordination group passes 0.075 Å from C(4) in I and 0.069 Å from C(4) in II.

Table 2. Bond lengths (Å) and angles (°) in phenylbis(selenourea)tellurium(II) chloride.

Te—Se(1)	2.7229(16)	Se(1)—Te—Se(2)	173.09(4)
Te—Se(2)	2.8895(17)	Se(1)—Te—C(1)	88.71(15)
Te—C(1)	2.129(6)	Se(2)—Te—C(1)	86.65(16)
C(1)—C(2)	1.391(10)	Te—C(1)—C(2)	119.2(4)
C(1)—C(6)	1.394(8)	Te—C(1)—C(6)	118.7(4)
C(2)—C(3)	1.408(12)	C(2)—C(1)—C(6)	122.0(6)
C(3)—C(4)	1.412(11)	C(1)—C(2)—C(3)	118.2(6)
C(4)—C(5)	1.358(12)	C(2)—C(3)—C(4)	120.4(7)
C(5)—C(6)	1.418(10)	C(3)—C(4)—C(5)	119.7(6)
Se(1)—C(7)	1.897(7)	C(4)—C(5)—C(6)	121.4(6)
Se(2)—C(8)	1.893(7)	C(5)—C(6)—C(1)	118.1(6)
C(7)—N(1)	1.306(7)	Te—Se(1)—C(7)	101.1(2)
C(7)—N(2)	1.321(9)	Te—Se(2)—C(8)	99.8(2)
C(8)—N(3)	1.309(8)	Se(1)—C(7)—N(1)	117.3(5)
C(8)—N(4)	1.313(10)	Se(1)—C(7)—N(2)	123.8(4)
		N(1)—C(7)—N(2)	118.8(6)
		Se(2)—C(8)—N(3)	120.5(5)
		Se(2)—C(8)—N(4)	118.4(4)
		N(3)—C(8)—N(4)	121.1(6)

Table 3. Bond lengths (Å) and angles (°) in phenylbis(thiourea)tellurium(II) chloride.^a

Te-S(1)	2.616(2)	S(1)-Te-S(2)	172.37(8)
Te-S(2)	2.766(3)	S(1)-Te-C(1)	87.9(2)
Te-C(1)	2.102(7)	S(2)-Te-C(1)	84.6(2)
Te-Cl	3.578(2)	C(1)-Te-Cl	163.8(2)
C(1)-C(2)	1.376(14)	Te-C(1)-C(2)	122.2(6)
C(1)-C(6)	1.423(14)	Te-C(1)-C(6)	117.6(6)
C(2)-C(3)	1.422(15)	C(2)-C(1)-C(6)	120.2(8)
C(3)-C(4)	1.38(2)	C(1)-C(2)-C(3)	121.3(9)
C(4)-C(5)	1.377(19)	C(2)-C(3)-C(4)	118.4(11)
C(5)-C(6)	1.421(15)	C(3)-C(4)-C(5)	121.3(10)
		C(4)-C(5)-C(6)	121.3(9)
		C(5)-C(6)-C(1)	117.4(9)
S(1)-C(7)	1.719(9)	Te-S(1)-C(7)	105.5(3)
S(2)-C(8)	1.706(9)	Te-S(2)-C(8)	105.0(3)
C(7)-N(1)	1.337(10)	S(1)-C(7)-N(1)	123.1(6)
C(7)-N(2)	1.328(12)	S(1)-C(7)-N(2)	118.0(6)
C(8)-N(3)	1.325(11)	N(1)-C(7)-N(2)	118.9(7)
C(8)-N(4)	1.341(12)	S(2)-C(8)-N(3)	118.9(6)
		S(2)-C(8)-N(4)	121.4(6)
		N(3)-C(8)-N(4)	119.7(8)

^a The C-H bond lengths vary from 0.94(9) Å to 1.18(9) Å, with an average value of 1.07 Å. The C-C-H bond angles vary from 111(5)° to 129(5)° with an average value of 120°.

The three-centre systems Se-Te-Se and S-Te-S are nearly linear, and the Te-C bond nearly bisects the angle of the three-centre system. Small deviations from linearity are also found in the structures of the phenyldithiocyanatotellurate(II) and the phenyldiselenocyanatotellurate(II) anions.³

The Se-Te-Se bonding system in I is slightly asymmetric. The average value of the two Te-Se bond lengths, 2.806 Å is, within the error, equal to the average Te-Se bond length found in the crystals of centrosymmetric square-planar com-

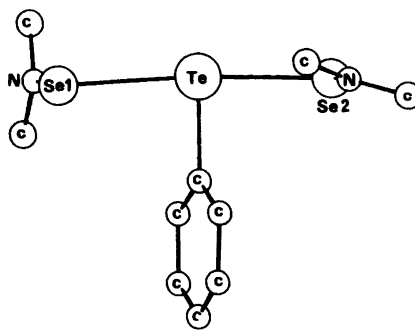


Fig. 1. Phenylbis(selenourea) tellurium(II) cation, as seen normal to the plane through the coordination group.

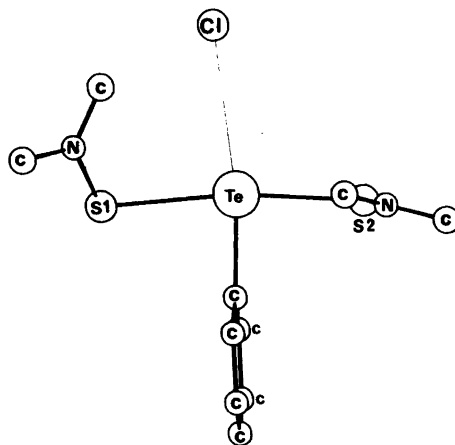


Fig. 2. Phenylbis(thiourea)tellurium(II) chloride, as seen normal to the plane through the coordination group.

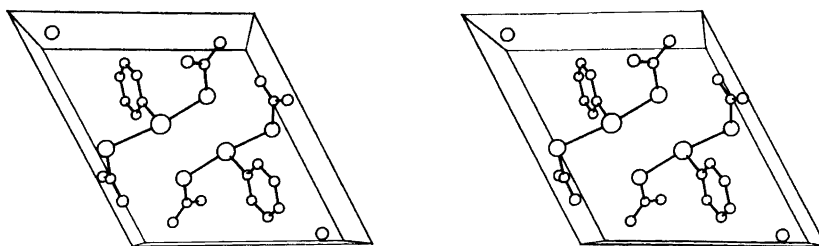


Fig. 3. A stereoscopic view of the content of the unit cell for phenylbis(selenourea)tellurium(II) chloride, as seen along the *c* crystal axis.

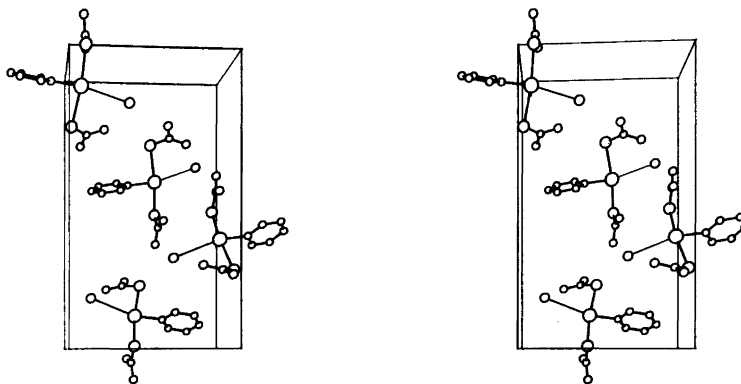


Fig. 4. A stereoscopic view of the content of the unit cell for phenylbis(thiourea)tellurium(II) chloride, as seen along the *c* crystal axis.

plexes of divalent tellurium,^{11–13} and about 0.02 Å longer than the average Te–Se bond length found in the structure of the phenyldiselenocyanatotellurate(II) anion.³

The two Te–S bond lengths indicate about the same asymmetry in the S–Te–S bonding as in the Se–Te–Se bonding. The average Te–S bond length, 2.691 Å is, within the error, equal to the average Te–S bond length found in the crystals of centrosymmetric square-planar complexes of divalent tellurium,^{14,15} and also equal to the average Te–S bond length found in the structure of the phenyldithiocyanatotellurate(II) anion.³

The Te–C bond lengths found in the two structures are equal within the accuracy of the structure determinations. The same Te–C bond length has been found in all structures where divalent tellurium is bonded to a phenyl group.^{1–6}

As pointed out by Foss *et al.*^{1,14,15} the coordination around the tellurium atom in the structure of II may be regarded as based on square-planar coordination, with the fourth position, *trans* to the phenyl group, virtually vacant. In II the chloride ion lies 0.84 Å from the least-squares plane through the coordination group. Thus, although at such a distance as to indicate only very weak bonding interaction, the chloride ion may be regarded as approaching the fourth coordination site of a square-planar arrangement.

In I the chloride ion is placed in a completely different position so that the tellurium atom is strictly three-coordinated. This is similar to what has been found in the structures of the phenyl-

dithiocyanato- and the phenyldiselenocyanatotellurate(II) anions.³

The atoms of the phenyltellurium group are nearly co-planar in each structure, the largest deviation from the least-squares plane through the phenyltellurium group is 0.020 Å in I and 0.037 Å in II. The angle between this plane and the least-squares plane through the coordination group is 72.74° in I and 88.45° in II.

The selenourea groups in I and the thiourea groups in II are nearly planar, the largest deviations being 0.019 Å in I and 0.080 Å in II. The angle between the two planes is 75.3° in I and 80.5° in II. The least-squares plane through the coordination group makes an angle of 87.5 and 77.5° with the least-squares plane through each of the two selenourea groups in I. The corresponding angles in II are 89.3 and 45.5°.

The Se–C bond lengths are, within the error, equal to the Se–C bond lengths found in the structures of bromo(ethyleneselenourea)phenyltellurium(II),⁵ ethyleneselenourea(iodo)phenyltellurium(II),⁶ and tetrakis(selenourea)tellurium(II) dichloride.¹² In II the S–C bond lengths are, within the error, equal to the S–C bond length found in the structures of chloro- and bromo(ethylene-thiourea)phenyltellurium(II).^{4,5} The Te–S–C bond angles are equal within the error of the structure determination.

Table 4. Nitrogen–chlorine distances (Å) and angles (°) in the structure of phenylbis(selenourea)-tellurium(II) chloride. x, y, z are the coordinates of the chloride ion given in Table 1.

Distance	Length N··Cl	Angle C–N··Cl	Distance from plane
N(1)··Cl($x, y-1, z+1$)	3.158(7)	110.6(5)	0.581
N(1)··Cl($2-x, 1-y, 1-z$)	3.357(8)	136.4(5)	1.540
N(2)··Cl($x, y-1, z+1$)	3.557(8)	91.1(3)	0.581
N(3)··Cl($x-1, y, z$)	3.326(6)	100.1(4)	-0.425
N(4)··Cl($x-1, y, z$)	3.363(6)	98.3(4)	-0.425
N(4)··Cl($1-x, 2-y, 1-z$)	3.436(8)	157.9(4)	1.059

Table 5. Nitrogen–chlorine distances (Å) and angles (°) in the structure of phenylbis(thiourea)-tellurium(II) chloride. x, y, z are the coordinates of the chloride ion given in Table 2.

Distance	Length N··Cl	Angle C–N··Cl	Distance from plane
N(1)··Cl($\frac{1}{2}-x, \bar{y}, z-\frac{1}{2}$)	3.256(8)	101.3(5)	-0.564
N(2)··Cl($\frac{1}{2}-x, \bar{y}, z-\frac{1}{2}$)	3.333(8)	97.9(6)	-0.564
N(2)··Cl($\frac{1}{2}-x, \bar{y}, \frac{1}{2}+z$)	3.306(8)	129.8(6)	1.151
N(3)··Cl($\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$)	3.255(8)	128.8(6)	0.290
N(4)··Cl(x, y, z)	3.227(9)	129.3(5)	0.269
N(4)··Cl($x, y, z-1$)	3.483(9)	108.5(5)	-1.465

HYDROGEN BONDING

Short intermolecular distances, probably involving hydrogen bonds, occur between amino nitrogen atoms and chloride ions. The nitrogen atoms are assumed to have a trigonal-planar bonding system, *i.e.*, the hydrogen atoms lie in or close to the planes through the selenourea groups in I, and the thiourea groups in II. The N··Cl distances, the N–C··Cl angles, and the distances of the chloride ions from the least-squares planes for I and II, are listed in Tables 4 and 5, respectively. The chloride ion, in both structures, takes part in six hydrogen bonds.

Hydrogen bonding to selenium, N–H··Se, has been found in crystals of selenourea¹⁶ and related compounds.^{12,17,18} In the crystals of I such hydrogen bonding probably occurs between N(2) (x, y, z) and Se(1') ($x, y, z+1$), and between N(3) (x, y, z) and Se(2') ($x, 1-y, 1-z$). The N(2)··Se(1') distance is 3.721(8) Å, and the N(3)··Se(2') distance is 3.614(6) Å. The C(7)–N(2)··Se(1') angle is 129.9(4)°, the C(8)–N(3)··Se(1') angle is 129.9(4)°, and the C(8)–N(3)··Se(2') angle is 145.0(4)°.

Hydrogen bonding to sulfur, N–H··S, has been found in crystals of thiourea.¹⁹ In the crystals of II

such hydrogen bonding probably occurs between N(1) (x, y, z) and S(1') ($x, y, z-1$), and between N(3) (x, y, z) and S(2') ($x, y, z-1$). The N(1)··S(1') distance is 3.407(4) Å, and the N(3)··S(2') distance is 3.515(7) Å. The C(7)–N(1)··S(1') angle is 169.7(5)°, and the C(8)–N(3)··S(2') angle is 149.1(5)°.

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Received June 8, 1978.