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The Crystal Structure of Tetrakis(selenourea)-tellurium(II) Dichloride

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The crystal structure of tetrakis(selenourea)tellurium(II) dichloride, $Te(su)_4Cl_2$, has been determined by X-ray methods, and refined by full-matrix least squares procedures. The crystals are triclinic, with $a = 7.921(4)$ Å, $b = 10.957(5)$ Å, $c = 5.940(3)$ Å, $\alpha = 90.25(4)°$, $\beta = 118.94(5)°$, and $\gamma = 96.15(4)°$.

In the salt, the tellurium atom of the $Te(su)_4^{2+}$ cation lies in a centre of symmetry, and is bonded to four selenium atoms in a square-planar arrangement, with Te–Se bond lengths, 2.814(3) and 2.809(3) Å. The bonds are thus equal within the error. Within the error, the Se–Te–Se angles are 90°.

Four-coordinated, square-planar complexes of tellurium(II) have been studied by Foss and co-workers [1] at this Institute. The crystal structures of complexes where tellurium(II) is bonded to four sulphur atoms, to two sulphur atoms and two halogen atoms, and to two sulphur atoms and two selenium atoms, have been determined.[1] The present work on tetrakis(selenourea)tellurium(II) dichloride, $Te(su)_4Cl_2$, where su = selenourea, has been carried out mainly for study of the $TeSe_4$ coordination group.

Tetrakis(selenourea)tellurium(II) dichloride was prepared by methods analogous to those used for the preparation of the corresponding thiourea complex.[2–4] Tellurium dioxide dissolved in hydrochloric acid reacts with a hydrochloric acid solution of four moles of selenourea, to give a red-brown solution from which orange crystals of the bis(selenourea) compound, $Te(su)_2Cl_2$, deposite:

$$Te^{IV} + 4\ su + 2\ Cl^- = Te(su)_2Cl_2 + (su)_2^{2+}$$

Tetravalent tellurium is reduced to the divalent state by two moles of selenourea, which thereby become oxidized to formamidinium diselenide. $Te(su)_2Cl_2$ dissolves in a solution of selenourea in hydrochloric acid, to give a yellow solution from which yellow crystals of $Te(su)_4Cl_2$ separate:

$$Te(su)_2Cl_2 + 2\ su = Te(su)_4^{2+} + 2\ Cl^-$$

Further preparative work on selenourea complexes of tellurium(II) will be made.

CRYSTAL DATA

Tetrakis(selenourea)tellurium(II) dichloride, $Te(su)_4Cl_2$, forms yellow, triclinic prisms, mainly elongated along the c axis, with $a = 7.921(4)$ Å, $b = 10.957(5)$ Å, $c = 5.940(3)$ Å, $\alpha = 90.25(4)°$, $\beta = 118.94(5)°$, an $\gamma = 96.15(4)°$. The unit cell dimensions were determined from zero-layer Weissenberg photographs around the three axes, using Cu$K\alpha$ radiation. 2θ-values were measured for 85 reflections and evaluated by means of a least squares program.

There is one formula unit per unit cell; density, calc. 2.56, found 2.54 g/cm^3. The space group, from subsequent structure analysis, is $P\bar{1}$ (No. 2). The crystals are isomorphous with those of the corresponding thiourea complex.[4,5]

Intensities were estimated visually from integrated Weissenberg photographs around the a and c axes, taken with Cu$K\alpha$ radiation using the multi-film technique. The three-dimensional refinement was based on the $0kl - 2kl$ and $hk0 - hk2$ data. 138 out of 141 $0kl$, 240 out of 248 $1kl$, 238 out of 244 $2kl$, 178 out of 185 $hk0$, 327 out of 335 $hk1$, and 318 out of 322 $hk2$ reflections, in all 1138 out of 1173 independent $0kl - 2kl$ and $hk0 - hk2$ reflections, accessible with Cu$K\alpha$ radiation, were observed with measurable intensities. Two different crystals were used; the crystal used for collection of the $0kl - 2kl$ data had the following dimensions, from an arbitrarily chosen origin to the crystal faces: to (100) and ($\bar{1}$00), 0.042 mm; to (010) and (0$\bar{1}$0), 0.048 mm; to (001) and (00$\bar{1}$), 0.065 mm. The dimensions of the crystal used for the $hk0 - hk2$ data were: to (120) and ($\bar{1}$20), 0.035 mm; to (010) and (0$\bar{1}$0), 0.018 mm; to (011), 0.069 mm; to (00$\bar{1}$), 0.075 mm. The linear absorption coefficient, $\mu = 245$ cm^{-1}.

The intensities were corrected for absorption by the method of Coppens *et al.*[6] A sub-division of 8, 6, and 12 Gaussian points along the a, b, and c axes respectively, was used for the crystal from which the $0kl - 2kl$ data were collected. The corresponding figures for the other crystal were 4, 8, and 16, respectively.

THE STRUCTURE ANALYSIS

Approximate x and y coordinates of the selenium atoms and the chlorine atom in the c-axis projection were found from the Patterson map. Tellurium lies in a centre of symmetry. A structure factor calculation based on these positions gave signs to most of the $hk0$ reflections. The carbon and nitrogen atoms were placed, in the c-axis projection, on the basis of subsequent Fourier maps.

The approximate z coordinates of the atoms were found from the structure of the isomorphous thiourea complex [5] and the known dimensions of the selenourea group.

The three-dimensional refinement was carried out on an IBM 360/50H computer, using a full-matrix least squares program minimizing the function, $r = \sum W(|F_o| - K|F_c|)^2$, where K is the scale factor, and $W = 1/[(Ka_1)^2 + (a_2F_o)^2/4W_o]$. The weight, W_o, is based on the estimated reliability of the film readings. The constants a_1 and a_2 were both given the value one. Unobserved reflections with calculated structure factors, $|F_c|$, greater than the threshold value, F_t, were included on the refinement with F_o equal to F_t.

Refinement with isotropic temperature factors brought the reliability index, R, to 0.14. The observed structure factors were then corrected for secondary extinction, using the method of Zachariasen,[7] neglecting the absorption term, since this correction had been carried out earlier: $F_{corr} = K F_o (1 + \beta C I_o)$, where $\beta = 2 (1 + \cos^4 2\theta)/(1 + \cos^2 2\theta)^2$, and C was found to be 6×10^{-6}. The intensities of reflections that occurred more than once in the data set were averaged, and such reflections thereafter included only once.

The final refinement based on the corrected observed structure factors, and with anisotropic temperature factors for tellurium and selenium, brought the reliability index, R, down to 0.089, with unobserved reflections included if $|F_c|$ exceeds the observable limit, F_t.

Most computer programs were made available by the Chemical Department of X-Ray Crystallography, Weizmann Institute of Science, Rehovoth, Israel, and modified for use on the IBM 360/50H computer by Dr. D. Rabinovich. The program used for extinction correction, and programs used for calculating distances and angles, and least squares planes, were written by Mr. K. Maartmann-Moe of this Institute.

The calculated structure factors were based on atomic scattering factors, given in the *International Tables* (Ref. 8, Table 3.3.1A). The scattering factors for tellurium and selenium were corrected for anomalous dispersion, real and imaginary parts (Ref. 8, Table 3.3.2A), by taking the amplitude of f as the corrected value.

The final atomic coordinates and temperature factors are listed in Tables 1 and 2, and the structure factors in Table 3.

Table 1. Atomic coordinates for tetrakis(selenourea)tellurium(II) dichloride, in fractions of triclinic cell edges. Isotropic temperature parameters (Å²) in the form $\exp[-8\pi^2 U(\sin^2\theta/\lambda^2)]$.
Standard deviations from least squares are given in parentheses.

	x	y	z	U
Te	0	0	0	
Se_1	−0.35821(31)	0.09579(17)	−0.2264(4)	
Se_2	0.14103(35)	0.17216(18)	−0.2374(4)	
C_1	−0.2736(30)	0.2070(18)	0.062(4)	0.054(4)
C_2	0.2575(28)	0.2957(17)	0.032(4)	0.049(4)
N_1	−0.3222(27)	0.1809(16)	0.234(4)	0.030(3)
N_2	−0.1753(28)	0.3154(17)	0.078(4)	0.034(4)
N_3	0.2994(29)	0.2793(17)	0.265(4)	0.048(4)
N_4	0.3104(30)	0.4076(18)	−0.030(4)	0.052(4)
Cl	−0.2331(9)	0.4547(4)	0.5486(11)	

Table 2. Anisotropic temperature parameters (Å²) in the form $\exp -[2\pi^2(h^2a^{-2}U_{11} + \cdots 2\,hka^{-1}b^{-1}U_{12} + \cdots]$. All values have been multiplied by 10^4.
Standard deviations are given in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Te	456(11)	228(6)	438(14)	−29(6)	−52(6)	266(12)
Se_1	488(12)	330(8)	430(16)	33(8)	−12(8)	236(15)
Se_2	642(13)	343(8)	484(17)	−15(8)	− 5(8)	298(16)
Cl	755(36)	330(19)	532(33)	−15(22)	−23(21)	388(41)

Table 3. Observed and calculated structure factors. Unobserved reflections are indicated by a minus sign on $F(O)$.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
1	0	0	40	42	2	-6	0	35	31	1	0	-1	30	40	3	-6	1	68	64	4	-3	-1	14	8
2	0	0	54	49	1	-6	0	107	103	2	0	-1	69	73	2	-6	1	20	17	5	-3	-1	29	25
3	0	0	49	46	0	6	0	40	35	3	0	-1	44	47	1	-6	1	62	62	6	-3	-1	5	2
4	0	0	19	16	1	6	0	60	54	4	0	-1	46	48	0	-6	1	46	43	7	-3	-1	9	6
5	0	0	21	20	2	6	0	32	30	5	0	-1	24	21	1	6	-1	22	20	8	-3	-1	31	29
6	0	0	35	35	3	6	0	34	31	6	0	-1	70	70	2	6	-1	28	23	9	-3	-1	14	12
7	0	0	11	10	4	6	0	24	23	7	0	-1	19	18	3	6	-1	12	8	7	4	1	5	4
8	0	0	26	23	5	6	0	20	19	8	0	-1	8	8	4	6	-1	20	19	6	4	1	32	29
8	-1	0	26	25	6	6	0	8	8	9	0	-1	10	10	5	6	-1	78	79	5	4	1	14	12
7	-1	0	8	6	7	6	0	32	33	8	-1	1	9	8	6	6	-1	23	20	4	4	1	32	31
6	-1	0	11	12	7	-7	0	16	15	7	-1	1	6	5	7	6	-1	-3	0	3	4	1	73	77
5	-1	0	16	18	6	-7	0	11	11	6	-1	1	20	18	7	-7	1	10	8	2	4	1	15	8
4	-1	0	34	40	5	-7	0	23	21	5	-1	1	38	37	6	-7	1	18	23	1	4	1	19	15
3	-1	0	52	63	4	-7	0	34	31	4	-1	1	44	51	5	-7	1	9	8	0	4	1	5	5
2	-1	0	112	127	3	-7	0	41	39	3	-1	1	25	26	4	-7	1	10	10	1	-4	-1	44	38
1	-1	0	33	36	2	-7	0	22	19	2	-1	1	72	83	3	-7	1	66	67	2	-4	-1	102	93
0	1	0	65	89	1	-7	0	70	71	1	-1	1	23	27	2	-7	1	24	21	3	-4	-1	94	85
1	1	0	29	26	0	7	0	40	33	0	-1	1	18	9	1	-7	1	15	14	4	-4	-1	31	27
2	1	0	8	10	1	7	0	23	17	1	1	-1	30	28	0	-7	1	28	23	5	-4	-1	49	47
3	1	0	46	47	2	7	0	18	16	2	1	-1	58	46	2	7	-1	36	34	6	-4	-1	9	8
4	1	0	13	10	3	7	0	30	27	3	1	-1	58	77	3	7	-1	70	64	7	-4	-1	7	6
5	1	0	26	26	4	7	0	29	27	4	1	-1	81	110	4	7	-1	26	23	8	-4	-1	22	23
6	1	0	63	63	5	7	0	45	46	5	1	-1	9	10	5	7	-1	38	36	7	5	1	4	4
7	1	0	11	11	6	7	0	12	13	6	1	-1	22	26	6	7	-1	12	12	6	5	1	8	7
8	1	0	20	7	7	-8	0	-3	1	7	1	-1	13	13	7	7	-1	3	1	5	5	1	16	14
8	-2	0	25	24	6	-8	0	17	14	8	1	-1	9	8	7	-8	1	9	8	4	5	1	51	49
7	-2	0	7	7	5	-8	0	6	4	9	1	-1	14	14	6	-8	1	14	14	3	5	1	73	74
6	-2	0	11	10	4	-8	0	30	28	8	-2	1	-2	0	5	-8	1	25	26	2	5	1	10	9
5	-2	0	41	45	3	-8	0	46	45	7	-2	1	17	16	4	-8	1	17	18	1	5	1	56	55
4	-2	0	6	4	2	-8	0	31	28	6	-2	1	-3	0	3	-8	1	48	47	0	5	1	18	14
3	-2	0	56	58	1	-8	0	72	66	5	-2	1	25	28	2	-8	1	5	3	1	-5	-1	36	35
2	-2	0	143	158	0	8	0	14	9	4	-2	1	48	55	1	-8	1	25	23	2	-5	-1	64	58
1	-2	0	8	2	1	8	0	52	45	3	-2	1	30	34	0	-8	1	40	37	3	-5	-1	26	22
0	2	0	24	23	2	8	0	50	47	2	-2	1	73	77	3	8	-1	68	65	4	-5	-1	33	30
1	2	0	32	30	3	8	0	36	32	1	-2	1	38	39	4	8	-1	13	12	5	-5	-1	75	71
2	2	0	66	61	4	8	0	20	22	0	-2	1	77	89	5	8	-1	16	16	6	-5	-1	10	9
3	2	0	55	60	5	8	0	38	37	1	2	-1	99	117	6	8	-1	9	7	7	-5	-1	5	3
4	2	0	72	75	6	8	0	9	6	2	2	-1	73	83	6	-9	1	18	18	8	-5	-1	10	9
5	2	0	30	29	6	-9	0	16	17	3	2	-1	42	48	5	-9	1	29	29	6	6	1	10	10
6	2	0	36	34	5	-9	0	12	11	4	2	-1	56	79	4	-9	1	9	9	5	6	1	12	8
7	2	0	-3	1	4	-9	0	33	30	5	2	-1	6	0	3	-9	1	15	13	4	6	1	26	23
8	2	0	-3	0	3	-9	0	55	50	6	2	-1	6	2	2	-9	1	21	19	3	6	1	9	7
8	-3	0	12	11	2	-9	0	11	8	7	2	-1	13	15	1	-9	1	14	12	2	6	1	35	33
7	-3	0	10	13	1	-9	0	7	6	8	2	-1	10	9	5	-10	1	21	23	1	6	1	124	121
6	-3	0	12	12	0	9	0	25	21	9	2	-1	16	18	4	-10	1	-3	0	0	6	1	24	19
5	-3	0	42	47	1	9	0	5	4	7	-3	1	21	20	3	-10	1	-3	4	1	-6	-1	24	23
4	-3	0	44	50	2	9	0	42	40	6	-3	1	17	17	2	-10	1	15	13	2	-6	-1	36	33
3	-3	0	43	45	3	9	0	34	33	5	-3	1	33	37	1	-10	1	9	8	3	-6	-1	14	10
2	-3	0	88	96	4	9	0	19	19	4	-3	1	47	55	5	-11	1	10	9	4	-6	-1	35	31
1	-3	0	34	38	5	9	0	32	33	3	-3	1	37	38	4	-11	1	5	2	5	-6	-1	52	46
0	3	0	38	37	6	-10	0	27	27	2	-3	1	9	4	3	-11	1	6	4	6	-6	-1	13	10
1	3	0	79	77	5	-10	0	19	18	1	-3	1	65	62	2	-11	1	30	43	7	-6	-1	35	32
2	3	0	25	25	4	-10	0	7	7	0	-3	1	10	8	7	1	1	11	9	8	-6	-1	11	10
3	3	0	57	63	3	-10	0	36	34	1	3	-1	103	98	6	1	1	10	7	6	7	1	11	10
4	3	0	60	63	2	-10	0	11	9	2	3	-1	85	89	5	1	1	35	32	5	7	1	9	6
5	3	0	11	8	1	-10	0	11	8	3	3	-1	14	14	4	1	1	26	24	4	7	1	22	19
6	3	0	24	24	0	10	0	39	36	4	3	-1	56	57	3	1	1	8	6	3	7	1	6	4
7	3	0	-4	1	1	10	0	15	14	5	3	-1	6	5	2	1	1	67	72	2	7	1	26	22
8	3	0	8	7	2	10	0	40	43	6	3	-1	20	19	1	1	1	56	49	1	7	1	69	66
7	-4	0	17	16	3	10	0	43	44	7	3	-1	28	31	0	1	1	94	132	0	7	1	30	25
6	-4	0	-4	0	4	10	0	5	7	8	3	-1	14	15	1	-1	-1	66	76	1	-7	-1	72	68
5	-4	0	61	66	6	-11	0	15	14	7	-4	1	29	25	2	-1	-1	32	39	2	-7	-1	37	36
4	-4	0	74	72	5	-11	0	21	21	6	-4	1	17	20	3	-1	-1	20	20	3	-7	-1	52	45
3	-4	0	53	35	4	-11	0	4	2	5	-4	1	-2	3	4	-1	-1	11	6	4	-7	-1	26	22
2	-4	0	16	17	3	-11	0	13	13	4	-4	1	35	38	5	-1	-1	30	27	5	-7	-1	33	30
1	-4	0	24	25	2	-11	0	5	0	3	-4	1	12	15	6	-1	-1	56	55	6	-7	-1	12	10
0	4	0	6	1	1	-11	0	5	2	2	-4	1	14	14	7	-1	-1	11	9	7	-7	-1	33	30
1	4	0	85	78	0	11	0	52	56	1	-4	1	60	71	8	-1	-1	18	20	8	-7	-1	9	8
2	4	0	52	50	1	11	0	26	27	0	-4	1	41	42	9	-1	-1	17	17	5	8	1	26	26
3	4	0	61	63	2	11	0	11	9	1	4	-1	121	108	7	2	1	6	6	4	8	1	21	20
4	4	0	56	57	3	11	0	24	23	2	4	-1	92	86	6	2	1	38	37	3	8	1	27	24
5	4	0	28	26	4	11	0	5	3	3	4	-1	47	44	5	2	1	42	39	2	8	1	21	17
6	4	0	-4	3	5	-12	0	12	12	4	4	-1	9	6	4	2	1	27	24	1	8	1	33	31
7	4	0	20	20	4	-12	0	17	15	5	4	-1	19	20	3	2	1	16	15	0	8	1	14	12
8	4	0	6	7	3	-12	0	10	11	6	4	-1	5	3	2	2	1	22	22	1	-8	-1	80	78
6	-5	0	-4	3	2	-12	0	30	35	7	4	-1	38	42	1	2	1	19	17	2	-8	-1	39	37
7	-5	0	31	38	1	-12	0	10	9	8	4	-1	18	19	0	2	1	80	96	3	-8	-1	5	2
6	-5	0	13	13	0	12	0	28	30	7	-5	1	19	18	1	-2	-1	55	53	4	-8	-1	39	35
5	-5	0	13	13	1	12	0	22	24	6	-5	1	20	21	2	-2	-1	91	98	5	-8	-1	14	10
4	-5	0	45	46	2	12	0	4	1	5	-5	1	16	18	3	-2	-1	85	84	6	-8	-1	-3	4
3	-5	0	15	16	3	12	0	11	11	4	-5	1	16	18	4	-2	-1	59	66	7	-8	-1	32	31
2	-5	0	12	8	4	-13	0	5	4	3	-5	1	14	11	5	-2	-1	9	7	8	-8	-1	10	10
1	-5	0	39	35	3	-13	0	5	4	2	-5	1	10	7	6	-2	-1	48	49	5	9	1	27	26
0	5	0	22	19	2	-13	0	29	30	1	-5	1	125	130	7	-2	-1	13	13	4	9	1	31	28
1	5	0	115	113	1	-13	0	15	15	0	-5	1	56	55	8	-2	-1	26	26	3	9	1	5	4
2	5	0	66	67	0	13	0	18	18	1	5	-1	32	22	9	-2	-1	16	14	2	9	1	21	21
3	5	0	7	8	1	13	0	13	13	2	5	-1	52	47	7	3	1	4	3	1	9	1	10	10
4	5	0	19	17	2	13	0	11	13	3	5	-1	4	4	6	3	1	38	37	0	9	1	9	6
5	5	0	6	5	3	0	1	19	18	4	5	-1	8	4	5	3	1	36	35	1	-9	-1	67	65
6	5	0	9	9	7	0	1	15	16	5	5	-1	38	38	4	3	1	24	20	2	-9	-1	37	36
7	5	0	26	26	6	0	1	10	6	6	5	-1	17	16	3	3	1	62	59	3	-9	-1	31	27
8	-6	0	-3	1	5	0	1	14	12	7	5	-1	33	34	2	3	1	45	42	4	-9	-1	32	28
7	-6	0	24	30	4	0	1	7	8	8	5	-1	19	20	1	3	1	45	43	5	-9	-1	15	11
6	-6	0	20	24	3	0	1	38	43	7	-6	1	7	7	0	3	1	101	122	6	-9	-1	13	13
5	-6	0	-4	2	2	0	1	109	118	6	-6	1	12	13	1	-3	-1	75	67	7	-9	-1	16	15
4	-6	0	16	15	1	0	1	34	34	5	-6	1	19	21	2	-3	-1	44	49	4	10	1	25	24
3	-6	0	24	22	0	0	1	59	75	4	-6	1	19	20	3	-3	-1	73	70	3	10	1	20	18

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
2	10	1	26	27	2	6	2	13	12	3	9	-2	4	1	7	-2	-2	31	30	0	7	5	13	14
1	10	1	5	3	2	7	2	31	28	3	8	-2	43	40	7	-3	-2	26	27	0	8	5	4	4
0	10	1	16	12	2	8	2	11	10	3	7	-2	69	61	7	-4	-2	12	12	0	-6	6	5	3
1	-10	-1	18	18	2	9	2	8	5	3	6	-2	103	90	7	-5	-2	9	5	0	-5	6	15	15
2	-10	-1	13	11	2	10	2	7	6	3	5	-2	52	46	7	-6	-2	14	11	0	-4	6	12	12
3	-10	-1	51	48	3	-12	2	14	14	3	4	-2	10	11	7	-7	-2	13	11	0	-3	6	15	15
4	-10	-1	49	47	3	-11	2	5	3	3	3	-2	38	40	7	-8	-2	26	25	0	-2	6	-3	0
5	-10	-1	6	4	3	-10	2	4	0	3	2	-2	28	20	7	-9	-2	34	32	0	-1	6	10	9
6	-10	-1	12	12	3	-9	2	12	9	3	1	-2	9	15	7	-10	-2	21	19	0	0	6	14	13
7	-10	-1	4	4	3	-8	2	7	4	3	0	-2	41	46	7	-11	-2	7	10	0	1	6	7	7
3	11	1	30	28	3	-7	2	24	23	3	-1	-2	23	19	8	7	-2	11	14	0	2	6	27	30
2	11	1	46	46	3	-6	2	48	49	3	-2	-2	16	13	8	6	-2	13	12	0	3	6	30	30
1	11	1	6	2	3	-5	2	65	70	3	-3	-2	46	46	8	5	-2	11	11	0	4	6	22	23
0	11	1	6	5	3	-4	2	30	28	3	-4	-2	73	70	8	4	-2	24	25	0	5	6	6	6
1	-11	-1	13	10	3	-3	2	21	19	3	-5	-2	123	116	8	3	-2	28	26	1	13	-1	13	16
2	-11	-1	4	0	3	-2	2	7	2	3	-6	-2	44	40	8	2	-2	32	31	1	12	-1	9	10
3	-11	-1	31	30	3	-1	2	14	10	3	-7	-2	19	16	8	1	-2	8	9	1	11	-1	28	31
4	-11	-1	16	16	3	0	2	7	7	3	-8	-2	33	29	8	0	-2	8	5	1	10	-1	44	48
5	-11	-1	14	12	3	1	2	23	22	3	-9	-2	8	5	8	-1	-2	16	18	1	9	-1	35	38
6	-11	-1	28	25	3	4	2	27	25	3	-10	-2	7	6	8	-2	-2	3	1	1	8	-1	10	10
2	12	1	19	21	3	5	2	45	45	3	-11	-2	18	21	8	-3	-2	15	16	1	7	-1	34	36
1	12	1	19	18	3	6	2	57	58	3	-12	-2	18	20	8	-4	-2	24	30	1	12	-3	7	6
0	12	1	36	39	3	7	2	35	34	3	-13	-2	13	14	8	-5	-2	21	22	1	11	-3	8	9
1	-12	-1	8	7	3	8	2	22	19	4	12	-2	21	22	8	-6	-2	16	16	1	10	-3	8	8
2	-12	-1	10	8	4	-11	2	21	22	4	11	-2	43	43	8	-7	-2	21	18	1	9	-3	12	9
3	-12	-1	16	15	4	-10	2	11	9	4	10	-2	26	26	8	-8	-2	13	12	1	8	-3	38	38
4	-12	-1	4	3	4	-9	2	4	0	4	9	-2	25	24	8	-9	-2	15	14	1	7	-3	67	68
5	-12	-1	17	17	4	-8	2	7	4	4	8	-2	8	6	9	4	-2	10	10	1	6	-3	77	80
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1	13	1	12	15	4	-6	2	6	6	4	6	-2	30	26	9	2	-2	10	10	1	4	-3	9	6
0	13	1	29	31	4	-5	2	12	10	4	5	-2	25	22	9	1	-2	11	10	1	3	-3	54	47
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2	-13	-1	11	12	4	-3	2	13	11	4	3	-2	30	28	9	-1	-2	12	10	1	1	-3	29	25
3	-13	-1	15	15	4	-2	2	35	35	4	2	-2	19	22	9	-2	-2	19	17	1	0	-3	44	41
4	-13	-1	7	7	4	-1	2	58	62	4	1	-2	45	56	9	-3	-2	21	18	1	-1	-3	23	21
5	-13	2	8	8	4	0	2	60	61	4	0	-2	112	133	9	-4	-2	21	29	1	-2	-3	28	23
0	-12	2	15	15	5	-9	2	13	14	4	-1	-2	77	79	9	-5	-2	10	11	1	-3	-3	46	46
0	-11	2	11	10	5	-8	2	23	26	4	-2	-2	47	43	9	-6	-2	-2	0	1	-4	-3	71	73
1	-10	2	6	4	5	-7	2	25	30	4	-3	-2	15	6	9	-7	-2	6	5	1	-5	-3	83	84
0	-9	2	41	40	5	-6	2	15	19	4	-4	-2	10	7	0	-13	1	12	13	1	-6	-3	34	29
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0	-7	2	51	48	5	-4	2	4	1	4	-6	-2	31	27	0	-11	1	15	16	1	-8	-3	29	28
0	-6	2	17	15	5	-3	2	14	13	4	-7	-2	28	24	0	-10	1	52	58	1	-9	-3	-3	1
0	-5	2	51	49	5	-2	2	10	7	4	-8	-2	7	4	0	-9	1	34	35	1	-10	-3	7	8
0	-4	2	70	71	6	-8	2	9	9	4	-9	-2	19	17	0	-12	3	14	16	1	-11	-3	32	33
0	-3	2	74	73	6	-7	2	9	8	4	-10	-2	25	24	0	-11	3	-3	3	1	11	-4	9	9
0	-2	2	42	35	6	-6	2	12	10	4	-11	-2	35	40	0	-10	3	-4	1	1	10	-4	4	5
0	-1	2	52	50	6	-5	2	9	7	4	-12	-2	20	25	0	-9	3	14	12	1	9	-4	13	14
0	0	2	34	28	6	-4	2	19	21	4	-13	-2	22	23	0	-8	3	17	17	1	8	-4	10	8
0	1	2	46	50	1	13	-2	7	7	5	11	-2	20	21	0	-7	3	23	22	1	7	-4	22	19
0	2	2	74	83	1	12	-2	12	11	5	10	-2	14	15	0	-6	3	28	25	1	6	-4	51	51
0	3	2	81	96	1	11	-2	10	8	5	9	-2	4	1	0	-5	3	7	4	1	5	-4	63	65
0	4	2	77	88	1	10	-2	26	27	5	8	-2	23	23	0	-4	3	41	41	1	4	-4	40	39
0	5	2	34	31	1	9	-2	54	53	5	7	-2	5	2	0	-3	3	64	63	1	3	-4	30	27
0	6	2	32	25	1	8	-2	64	59	5	6	-2	29	26	0	-2	3	75	80	1	2	-4	9	10
0	7	2	24	21	1	7	-2	56	50	5	5	-2	66	66	0	-1	3	61	65	1	1	-4	15	13
0	8	2	36	32	1	6	-2	25	20	5	4	-2	56	57	0	0	3	9	5	1	0	-4	7	3
0	9	2	31	29	1	5	-2	33	31	5	3	-2	53	60	0	1	3	32	33	1	-1	-4	36	34
0	10	2	-2	3	1	4	-2	28	32	5	2	-2	4	4	0	2	3	47	46	1	-2	-4	9	5
0	11	2	13	10	1	3	-2	42	46	5	1	-2	15	16	0	3	3	35	32	1	-3	-4	6	1
0	12	2	6	6	1	2	-2	51	57	5	0	-2	29	26	0	4	3	33	32	1	-4	-4	25	21
1	-13	2	16	17	1	1	-2	90	92	5	-1	-2	24	25	0	5	3	60	61	1	-5	-4	46	44
1	-12	2	10	10	1	0	-2	22	22	5	-2	-2	10	9	0	6	3	25	24	1	-6	-4	59	64
1	-11	2	25	28	1	-1	-2	49	53	5	-3	-2	18	18	0	7	3	28	27	1	-7	-4	41	40
1	-10	2	33	31	1	-2	-2	68	76	5	-4	-2	11	9	0	8	3	28	28	1	-8	-4	26	27
1	-9	2	22	20	1	-3	-2	83	93	5	-5	-2	12	9	0	9	3	31	33	1	-9	-4	14	12
1	-8	2	7	6	1	-4	-2	62	67	5	-6	-2	66	68	0	10	3	25	28	1	10	-5	9	11
1	-7	2	35	35	1	-5	-2	20	17	5	-7	-2	57	59	0	11	3	4	4	1	9	-5	13	14
1	-6	2	10	9	1	-6	-2	17	19	5	-8	-2	47	43	0	-11	4	29	29	1	8	-5	7	5
1	-5	2	16	17	1	-7	-2	39	36	5	-9	-2	7	7	0	-10	4	13	13	1	7	-5	13	13
1	-4	2	86	90	1	-8	-2	29	28	5	-10	-2	-2	1	0	-9	4	-3	0	1	6	-5	9	8
1	-3	2	74	76	1	-9	-2	31	32	5	-11	-2	3	2	0	-8	4	14	10	1	5	-5	14	13
1	-2	2	58	59	1	-10	-2	43	48	5	-12	-2	5	5	0	-7	4	21	20	1	4	-5	51	51
1	-1	2	16	16	1	-11	-2	27	29	6	10	-2	24	26	0	-6	4	6	5	1	3	-5	46	44
1	0	2	54	52	1	-12	-2	18	18	6	9	-2	25	24	0	-5	4	21	17	1	2	-5	41	37
1	1	2	37	37	1	-13	-2	13	13	6	8	-2	27	28	0	-4	4	6	4	1	1	-5	-3	1
1	2	2	40	42	2	13	-2	24	30	6	7	-2	14	14	0	-3	4	6	3	1	0	-5	16	15
1	3	2	6	3	2	12	-2	29	34	6	6	-2	19	17	0	-2	4	30	29	1	-1	-5	16	15
1	4	2	26	28	2	11	-2	8	6	6	5	-2	31	32	0	-1	4	69	72	1	-2	-5	25	24
1	5	2	10	9	2	10	-2	6	4	6	4	-2	29	28	0	0	4	79	81	1	-3	-5	7	1
1	6	2	13	13	2	9	-2	21	18	6	3	-2	21	22	0	1	4	25	24	1	-4	-5	8	5
1	7	2	67	66	2	8	-2	34	29	6	2	-2	22	21	0	2	4	-4	0	1	-5	-5	12	10
1	8	2	45	49	2	7	-2	24	19	6	1	-2	15	18	0	3	4	25	25	1	-6	-5	13	12
1	9	2	51	52	2	6	-2	36	31	6	0	-2	8	8	0	4	4	-5	0	1	-7	-5	39	41
1	10	2	11	10	2	5	-2	12	8	6	-1	-2	53	51	0	5	4	10	10	1	-8	-5	35	36
1	11	2	8	7	2	4	-2	21	18	6	-2	-2	60	65	0	6	4	32	33	1	-9	-5	21	26
1	12	2	5	4	2	3	-2	86	82	6	-3	-2	57	58	0	7	4	14	13	1	6	-6	10	9
2	-13	2	8	9	2	2	-2	101	108	6	-4	-2	25	24	0	8	4	15	15	1	5	-6	15	13
2	-12	2	10	8	2	1	-2	105	132	6	-5	-2	21	19	0	9	4	14	14	1	4	-6	-2	1
2	-11	2	23	22	2	0	-2	10	4	6	-6	-2	13	10	0	10	4	26	26	1	3	-6	22	23
2	-10	2	36	38	2	-1	-2	7	0	6	-7	-2	11	9	0	-9	5	13	13	1	2	-6	24	24
2	-9	2	31	30	2	-2	-2	63	63	6	-8	-2	16	15	0	-8	5	15	12	1	1	-6	26	27
2	-8	2	30	28	2	-3	-2	12	9	6	-9	-2	-3	1	0	-7	5	6	4	1	0	-6	7	7
2	-7	2	5	2	2	-4	-2	43	39	6	-10	-2	-3	0	0	-6	5	14	14	1	-1	-6	23	23
2	-6	2	33	33	2	-5	-2	76	74	6	-11	-2	-2	1	0	-5	5	11	10	1	-2	-6	18	16
2	-5	2	27	24	2	-6	-2	38	37	6	-12	-2	14	20	0	-4	5	13	13	1	-3	-6	26	25
2	-4	2	26	23	2	-7	-2	30	28	7	8	-2	18	19	0	-3	5	20	18	1	-4	-6	6	7
2	-3	2	15	15	2	-8	-2	38	36	7	7	-2	10	8	0	-2	5	8	6	1	-5	-6	8	6
2	-2	2	32	30	2	-9	-2	36	36	7	6	-2	15	13	0	-1	5	13	10	1	-6	-6	8	9
2	-1	2	6	1	2	-10	-2	36	44	7	5	-2	7	7	0	0	5	36	36	1	11	3	11	13
2	0	2	43	44	2	-11	-2	18	19	7	4	-2	27	27	0	1	5	48	49	1	10	3	34	41
2	1	2	112	119	2	-12	-2	8	4	7	3	-2	27	26	0	2	5	37	39	1	9	3	36	36
2	2	2	67	70	2	-13	-2	18	18	7	2	-2	37	39	0	3	5	27	25	1	8	3	23	24
2	3	2	45	48	3	12	-2	11	12	7	1	-2	28	27	0	4	5	12	9	1	7	3	16	16
2	4	2	10	10	3	11	-2	6	5	7	0	-2	17	18	0	5	5	6	6	1	6	3	18	17
2	5	2	4	2	3	10	-2	7	6	7	-1	-2	24	29	0	6	5	-3	2	1	5	3	15	13

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
1	4	3	23	25	1	0	5	5	6	2	4	4	27	28	2	-1	-3	60	66	2	5	-5	33	32
1	3	3	54	56	1	-1	5	8	8	2	3	4	17	19	2	-2	-3	42	41	2	4	-5	28	25
1	2	3	42	44	1	-2	5	14	13	2	2	4	16	16	2	-3	-3	45	48	2	3	-5	44	42
1	1	3	31	29	1	-3	5	-3	3	2	1	4	16	16	2	-4	-3	7	3	2	2	-5	9	5
1	0	3	25	24	1	-4	5	19	19	2	0	4	14	12	2	-5	-3	8	7	2	1	-5	30	26
1	-1	3	63	64	1	-5	5	35	38	2	-1	4	17	18	2	-6	-3	34	33	2	0	-5	22	22
1	-2	3	34	35	1	-6	5	28	32	2	-2	4	44	45	2	-7	-3	22	20	2	-1	-5	11	6
1	-3	3	48	48	1	-7	5	11	11	2	-3	4	35	36	2	-8	-3	14	13	2	-2	-5	49	47
1	-4	3	10	11	1	2	6	8	10	2	-4	4	24	23	2	-9	-3	21	21	2	-3	-5	37	38
1	-5	3	25	24	1	1	6	-2	3	2	-5	4	-5	3	2	-10	-3	28	29	2	-4	-5	55	53
1	-6	3	27	25	1	0	6	9	9	2	-6	4	19	21	2	-11	-3	42	45	2	-5	-5	17	16
1	-7	3	17	16	1	-1	6	3	3	2	-7	4	9	10	2	11	-4	13	14	2	-6	-5	18	16
1	-8	3	43	44	1	-2	6	12	11	2	4	5	6	5	2	10	-4	30	36	2	-7	-5	11	11
1	5	4	10	10	1	-3	6	17	18	2	3	5	13	11	2	9	-4	30	30	2	-8	-5	20	19
1	8	4	15	18	1	-4	6	21	23	2	2	5	7	6	2	8	-4	32	30	2	-9	-5	6	7
1	7	4	9	7	2	11	2	17	17	2	1	5	12	11	2	7	-4	9	6	2	8	-6	11	11
1	6	4	13	11	2	9	3	7	9	2	0	5	24	26	2	6	-4	28	25	2	7	-6	8	9
1	5	4	37	39	2	8	3	17	18	2	-1	5	31	33	2	5	-4	29	27	2	6	-6	19	19
1	4	4	43	44	2	7	3	13	11	2	-2	5	19	19	2	4	-4	27	24	2	5	-6	6	5
1	3	4	31	31	2	6	3	24	21	2	-3	5	16	16	2	3	-4	7	5	2	4	-6	19	18
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1	1	4	6	4	2	4	3	51	50	2	12	-1	11	11	2	1	-4	13	10	2	2	-6	37	38
1	0	4	33	32	2	3	3	50	49	2	11	-1	5	4	2	0	-4	26	26	2	1	-6	22	20
1	-1	4	22	20	2	2	3	57	56	2	10	-1	9	8	2	-1	-4	98	100	2	0	-6	-5	3
1	-2	4	10	11	2	1	3	13	12	2	9	-1	34	35	2	-2	-4	78	79	2	-1	-6	15	14
1	-3	4	21	20	2	0	3	26	24	2	8	-1	36	37	2	-3	-4	50	46	2	-2	-6	19	19
1	-4	4	10	9	2	-1	3	34	29	2	12	-3	26	30	2	-4	-4	7	3	2	-3	-6	10	10
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1	-6	4	27	29	2	-3	3	32	33	2	10	-3	13	14	2	-6	-4	-6	0	2	-5	-6	33	34
1	-7	4	37	40	2	-4	3	31	31	2	9	-3	14	14	2	-7	-4	25	24	2	-6	-6	13	13
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1	7	5	17	18	2	-6	3	17	17	2	7	-3	19	17	2	-9	-4	-5	3	2	4	-7	5	4
1	6	5	31	34	2	-7	3	25	24	2	6	-3	14	13	2	-10	-4	5	4	2	3	-7	10	10
1	5	5	33	34	2	-8	3	22	21	2	5	-3	27	26	2	-11	-4	14	13	2	2	-7	13	13
1	4	5	11	10	2	8	4	11	13	2	4	-3	15	14	2	10	-5	6	5	2	1	-7	30	31
1	3	5	4	4	2	7	4	15	15	2	3	-3	8	6	2	9	-5	25	27	2	0	-7	26	27
1	2	5	-3	2	2	6	4	14	13	2	2	-3	44	42	2	8	-5	19	20	2	-1	-7	12	11
1	1	5	17	16	2	5	4	32	34	2	1	-3	83	86	2	7	-5	19	19	2	-2	-7	6	4
										2	0	-3	99	117	2	6	-5	11	9	2	-3	-7	11	10

THE TETRAKIS(SELENOUREA)TELLURIUM(II) ION

Bond lengths and angles in the tetrakis(selenourea)tellurium(II) ion, based on the atomic coordinates in Table 1, are listed in Table 4. The uncertainties in cell dimensions are taken into account in the given standard deviations.

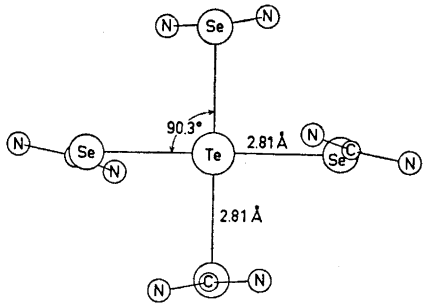

Fig. 1. The tetrakis(selenourea)tellurium(II) ion in the dichloride salt, as seen normal to the $TeSe_4$ plane.

With tellurium in a centre of symmetry, the $TeSe_4$ grouping is exactly planar. The Se – Te – Se angles are 90° within the error, and the Te – Se bonds, 2.814(3) and 2.809(3) Å, are equal within the error. The Te – Se bonds are 0.27 Å longer than the sum of the single covalent radii of tellurium and selenium, 2.54 Å. The same Te – Se bond length, 2.81 Å, has been found in *trans*-bis(selenocyanato)bis(ethylenethiourea)tellurium(II), $Te(etu)_2(SeCN)_2$.[1] With single covalent bond radii for ligands, 1.17 Å for selenium in this instance,

Table 4. Dimensions of the tetrakis(selenourea)tellurium(II) ion. Bond lengths (Å) and angles (°). Standard deviations are given in parentheses.

$Te-Se_1 = 2.814(3)$	$\angle Se_1-Te-Se_2 = 90.27(10)$
$Te-Se_2 = 2.809(4)$	$\angle Te-Se_1-C_1 = 91.7(7)$
	$\angle Te-Se_2-C_2 = 98.0(8)$
$Se_1-C_1 = 1.889(25)$	$\angle Se_1-C_1-N_1 = 119.8(13)$
$C_1-N_1 = 1.27(4)$	$\angle Se_1-C_1-N_2 = 120.1(18)$
$C_1-N_2 = 1.33(3)$	$\angle N_1-C_1-N_2 = 119.9(20)$
$Se_2-C_2 = 1.879(19)$	$\angle Se_2-C_2-N_3 = 124.7(13)$
$C_2-N_3 = 1.27(3)$	$\angle Se_2-C_2-N_4 = 115.7(15)$
$C_2-N_4 = 1.36(3)$	$\angle N_3-C_2-N_4 = 119.5(17)$

Table 5. Distances of atoms in the selenourea groups from the least squares planes of the groups. The equations of the planes were calculated with the selenium coordinates given six times the weight of the carbon and nitrogen coordinates, and refer to the axes of the unit cell, with coordinates X, Y, and Z in Å.

Plane through Se_1, C_1, N_1, N_2:

$$0.7439\,X - 0.5251\,Y + 0.0982\,Z + 2.7929 = 0$$

Se_1	−0.001 Å	C_1	0.027 Å	N_1	−0.010 Å
				N_2	−0.009

Plane through Se_2, C_2, N_3, N_4:

$$0.9379\,X - 0.3664\,Y - 0.2471\,Z - 0.7038 = 0$$

Se_2	−0.001 Å	C_2	−0.025 Å	N_3	0.010 Å
				N_4	0.009

the bond lengths correspond to a bonding radius of 1.64 Å for tellurium(II) in centrosymmetric, square-planar complexes.[1]

Bond lengths and angles in the selenourea groups do not deviate significantly from the values found in the crystals of selenourea.[10]

Each of the selenourea groups is planar within the error, the largest deviation of an atom from the least squares planes being 0.03 Å (*cf.* Table 5).

The angle between the Te–Se–C plane and the $TeSe_4$ plane is 91° for the Se_1 selenourea group, and 93° for the Se_2 selenourea group. The angles between the Te–Se–C plane and the least squares planes of the selenourea groups are 84° and 15°, respectively, for the Se_1 and Se_2 selenourea groups. The angle between the least squares planes of the Se_1 and Se_2 selenourea groups is 22°. All these angles are nearly the same as found in the isomorphous crystals of the corresponding thiourea complex.[5]

THE PACKING IN THE CRYSTALS

There is in the crystals a close intermolecular selenium-selenium contact of 3.447(3) Å, between Se_1 and $Se_1'(\bar{x}-1,\bar{y},\bar{z}-1)$. The $Te-Se_1-Se_1'$ angle is 103.31(8)°, and the $C_1-Se_1-Se_1'$ angle is 163.5(8)°. Se_1' lies 0.412 Å out of the $Te-Se_1-C_1$ plane.

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. The N···Cl distances, the C–N···Cl angles, and the distances of the chloride ions from the least squares planes of the selenourea groups are listed in Table 6. In the

Table 6. Nitrogen-chlorine distances (Å) and angles (°). Standard deviations are given in parentheses.

Distance	Length N···Cl	Angle C–N···Cl	Distance from plane
$N_1\cdots Cl_A$	3.350(19)	102.3(12)	−0.877
$N_2\cdots Cl_A$	3.421(26)	97.6(17)	−0.877
$N_2\cdots Cl_B$	3.351(24)	121.4(13)	−1.461
$N_2\cdots Cl_D$	3.604(17)	149.2(12)	1.292
$N_3\cdots Cl_C$	3.557(20)	95.8(13)	2.363
$N_3\cdots Cl_D$	3.295(23)	102.0(14)	−1.824
$N_4\cdots Cl_C$	3.564(18)	93.7(11)	2.363
$N_4\cdots Cl_D$	3.559(29)	88.3(17)	−1.824
$N_4\cdots Cl_E$	3.250(25)	137.7(13)	−0.357

table, Cl_A denotes a chloride ion at (x,y,z), Cl_B at $(x,y,z-1)$, Cl_C at $(1+x,y,z)$, Cl_D at $(\bar{x},1-y,1-z)$, and Cl_E at $(\bar{x},1-y,\bar{z})$, where x,y,z are the Cl coordinates of Table 1.

The shape of the N–H···Cl bonding systems, and the N···Cl distances, differ very little from what has been found in the crystal structure of the isomorphous thiourea compound.[5] The N···Cl distances are in this work found a little longer, about 0.06 Å in average.

Since Cl_C and Cl_D are rather far out of the selenourea plane, the N_3–H and N_4–H bonds are probably directed not toward Cl_C and Cl_D, but toward their midpoint, the symmetry centre at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$. This symmetry centre is 0.27 Å out of the selenourea plane, 2.71 Å from N_3 and 2.87 Å from N_4, at angles of 101° and 91° with the C–N bonds.

Hydrogen bonding to selenium, N–H···Se, has been found in crystals of selenourea [10] and related compounds.[11,12] In the crystal structure reported here, such hydrogen bonding probably occurs between N_1 and Se_1'' $(\bar{x}-1,\bar{y},\bar{z})$. The other hydrogen atom of N_1 bonds to Cl_A. Cl_A and Se_1'' are −0.877 and −0.306 Å, respectively, out of the Se_1 selenourea plane, the $N_1\cdots Cl_A$ distance is 3.350 Å, and the $N_1\cdots Se_1''$ distance is 3.722 Å. The C–N···Cl angle is 102.3°, the C–N···Se angle is 126.9°, and the Cl···N···Se angle is 127.5°.

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