

The Crystal Structure of Trithioureatellurium(II) Hydrogendifluoride

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The crystals of the salt, $\text{Te}(\text{tu})_3(\text{HF}_2)_2$ where tu = thiourea, have been shown by X-ray analysis to contain binuclear cations, $[\text{Te}(\text{tu})_3]_2^{4+}$, based on four-coordinated tellurium(II) in a planar, distorted square arrangement. The crystals are monoclinic, space group $C2h^5 - P2_1/c$ with two centrosymmetric $[\text{Te}(\text{tu})_3]_2(\text{HF}_2)_4$ units per unit cell, and $a = 5.91 \text{ \AA}$, $b = 20.68 \text{ \AA}$, $c = 11.47 \text{ \AA}$, $\beta = 95^\circ$.

The derived bond lengths and angles are, $\text{Te}-\text{S}_1 = 2.53 \text{ \AA}$, $\text{Te}-\text{S}_2 = 2.47 \text{ \AA}$, $\text{Te}-\text{S}_3 = 2.86 \text{ \AA}$, $\text{Te}-\text{S}_3' = 3.02 \text{ \AA}$, each $\pm 0.015 \text{ \AA}$, where S_3 and S_3' are the bridging sulphur atoms and are *trans* to S_1 and S_2 , respectively; $\angle \text{S}_1-\text{Te}-\text{S}_2 = 95.4^\circ$, $\angle \text{S}_2-\text{Te}-\text{S}_3 = 89.8^\circ$, $\angle \text{S}_3-\text{Te}-\text{S}_3' = 82.8^\circ$, $\angle \text{S}_3'-\text{Te}-\text{S}_1 = 92.1^\circ$, each $\pm 0.4^\circ$. A least-squares plane through the two tellurium and six sulphur atoms of a binuclear cation passes 0.02 \AA from the tellurium atoms and $0.008-0.025 \text{ \AA}$ from the sulphur atoms. The mean length of *trans*-positioned Te-S bonds, 2.70 \AA for $\text{S}_1-\text{Te}-\text{S}_3$ and 2.75 \AA for $\text{S}_2-\text{Te}-\text{S}_3'$, is only in the case of the latter, more asymmetric pair significantly larger than the Te-S bond length in the centrosymmetric, square-planar $\text{Te}(\text{tu})_4^{2+}$ cation, $2.68-2.69 \text{ \AA}$.

Recent preparative and X-ray crystallographic studies of complexes of divalent tellurium have led to the isolation and characterization of salts of cations TeL_4^{2+} with L = thiourea,¹ ethylenethiourea,² or propylenethiourea.³ The crystal structures of two salts, tetrathioureatellurium(II) dichloride and the corresponding dihydrate, have been determined.⁴ Only a few examples of salts containing three instead of four thiourea or substituted thiourea ligands per tellurium atom were encountered: trithioureatellurium(II) hydrogendifluoride,⁵ tris(ethylenethiourea)tellurium(II) perchlorate,² and tris(propylenethiourea)tellurium(II) perchlorate,³ the latter occurs in two crystalline forms. The crystal structure of the former of these salts is described here; the results reveal a binuclear cation based on four-coordinated, square-planar tellurium(II).

CRYSTAL DATA

The salt, $\text{Te}(\text{tu})_3(\text{HF}_2)_2$, forms yellow, monoclinic prisms $\{011\}$ with $a = 5.91 \text{ \AA}$, $b = 20.68 \text{ \AA}$, $c = 11.47 \text{ \AA}$, $\beta = 95^\circ$, and four formula units per unit cell. The space group, from systematic absences, is $C_{2h}^5 - P2_1/c$.

Intensities were estimated visually from zero-layer Weissenberg photographs around the a and c axes, taken with $\text{CuK}\alpha$ radiation. Small crystals were used in order to minimize absorption; the c -axis photographs were integrated. Of 296 $0kl$ reflections and 160 hkl reflections accessible with $\text{CuK}\alpha$ radiation, 195 and 109, respectively, were observed with measurable intensities.

The calculated structure factors were based on the Thomas-Fermi scattering curve for tellurium, that of Viervoll and Ögrim⁶ for sulphur, and those of Berghuis *et al.*⁷ for fluoride ion, carbon and nitrogen. Most of the calculations were made on the IBM 650 computer, using Shiono's programs.⁸ Summations were made at 12° , 3° , and 6° intervals, respectively, along the a , b , and c axes.

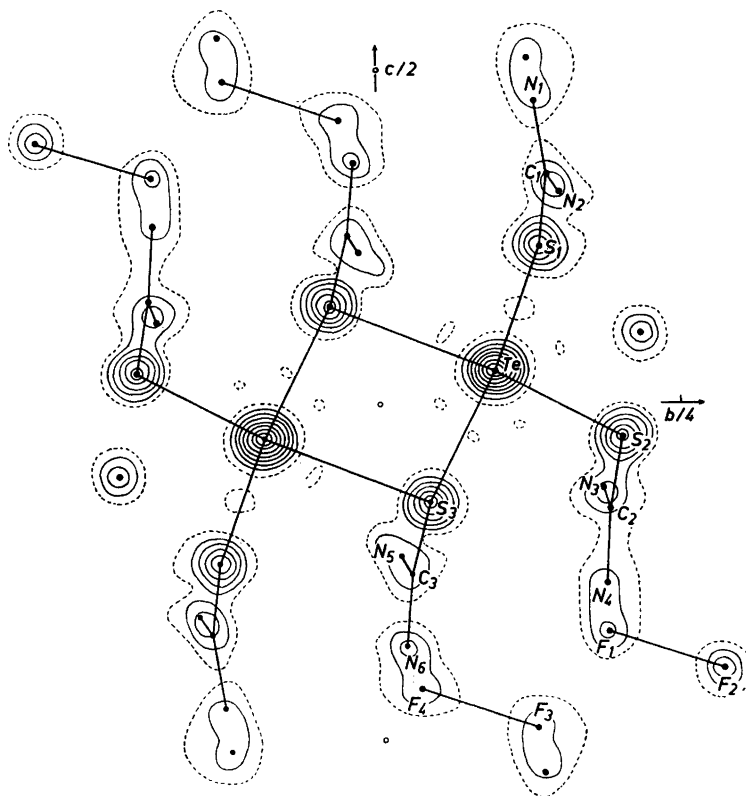


Fig. 1. Electron density of trithioureatellurium(II) hydrogendifluoride along the a axis. The 4-electron line is dashed. Contour intervals: $12 \text{ e}\cdot\text{\AA}^{-2}$ for tellurium, $4 \text{ e}\cdot\text{\AA}^{-2}$ for the other atoms.

THE STRUCTURE ANALYSIS

The projection along the a axis was solved and refined first. The approximate y and z coordinates of tellurium were found from Patterson line sections at $v = \frac{1}{2}$ and $w = \frac{1}{2}$, and a Fourier synthesis of 114 of the strongest $0kl$ reflections, with signs based on the tellurium contributions, gave the sulphur positions. The carbon and nitrogen atoms were placed partly on the basis of subsequent Fourier maps, and partly with reference to the known dimensions of the thiourea group. Refinement was carried out by repeated difference syntheses. The final $0kl$ Fourier map is shown in Fig. 1.

The x coordinates were determined from the $hk0$ data. A Patterson line section at $v = \frac{1}{2}$ gave the approximate x coordinate of tellurium, and work then proceeded as for the a -axis projection, with successive Fourier and difference syntheses.

The positive direction of the a axis relative to the c axis was established through comparison of calculated and observed values of selected $h0l$ and $h0\bar{l}$ reflections.

Table 1. Atomic coordinates for trithioureate tellurium(II) hydrogendifluoride, in fractions of monoclinic cell edges. Origin at a centre of symmetry.

	x	y	z
Te	-0.1274	0.0953	0.0500
S ₁	-0.2870	0.1352	0.2348
C ₁	-0.061	0.142	0.341
N ₁	-0.109	0.131	0.449
N ₂	0.160	0.151	0.313
S ₂	-0.1522	0.2008	-0.0510
C ₂	-0.385	0.192	-0.158
N ₃	-0.596	0.185	-0.127
N ₄	-0.330	0.185	-0.268
S ₃	0.0700	0.0401	-0.1458
C ₃	-0.156	0.024	-0.251
N ₅	-0.370	0.016	-0.226
N ₆	-0.110	0.019	-0.362
F ₁	0.133	0.187	-0.339
F ₂	0.278	0.283	-0.397
F ₃	0.476	0.128	-0.484
F ₄	0.327	0.032	-0.424

The final atomic coordinates are listed in Table 1, and observed and calculated $0kl$ and $hk0$ structure factors in Table 2. The $0kl$ reflections marked with an asterisk in Table 2, were omitted from the last difference syntheses and calculations of scale factors, and were included in the final Fourier map with their calculated instead of observed values; they are those with I_o above a certain value and most of them are probably subject to extinction. In the temperature factor $\exp[-B(\sin^2\theta/\lambda^2)]$ applied to the calculated structure factors, the values of B are, in \AA^2 units: $B = 2.8$ for sulphur, 3.5 for carbon and fluorine, and 3.3 for nitrogen in the $0kl$ zone; $B = 2.1$ for sulphur, 2.5 for carbon and nitrogen, and 3.0 for fluorine in the $hk0$ zone. For tellurium,

Table 2. Observed and calculated $0kl$ and $hk0$ structure factors for trithioureatellurium(II) hydrogendifluoride.

k	F_o	F_c	k	F_o	F_c	k	F_o	F_c
	$0kl$ zone							
	$0k0$		10	116	+ 125	10	33	+ 33
2	46	+ 46	11	29	+ 28	11	< 14	+ 14
4	113*	- 132	12	31	+ 29	12	< 20	+ 19
6	44	+ 20	13	81	+ 76	13	96	+ 100
8	< 10	+ 3	14	32	- 34	14	< 16	+ 22
10	133*	+ 132	15	38	+ 41	15	42	+ 44
12	30	- 40	16	64	- 64	16	< 17	- 6
14	50	- 50	17	26	- 22	17	65	- 73
16	74	- 67	18	< 17	0	18	< 17	- 18
18	64	- 78	19	< 17	0	19	41	- 44
20	49	+ 44	20	41	+ 44	20	< 16	+ 4
22	38	+ 53	21	< 16	- 4	21	< 15	- 9
24	< 13	- 7	22	< 15	+ 11	22	< 14	+ 8
26	23	- 28	23	22	+ 27	23	34	+ 31
			24	< 13	+ 7	24	< 11	- 4
			25	11	+ 14	25	14	+ 14
	$0k1$							
1	87*	+ 120		$0k3$			$0k5$	
2	43	+ 45	1	33	+ 42	1	< 11	- 1
3	56	- 58	2	53	+ 64	2	139	+ 157
4	16	+ 16	3	55	- 57	3	69	+ 66
5	74	- 76	4	87	+ 93	4	104	+ 110
6	182*	- 214	5	52	- 57	5	< 12	- 14
7	165*	- 189	6	61	- 58	6	68	- 72
8	77	- 83	7	54	- 51	7	25	+ 23
9	82	+ 83	8	128*	- 146	8	77	- 84
10	19	+ 10	9	46	+ 40	9	< 14	+ 2
11	113	+ 127	10	45	- 44	10	26	- 24
12	83	- 85	11	106	+ 112	11	< 15	+ 1
13	15	- 11	12	59	+ 63	12	61	+ 58
14	35	+ 27	13	29	+ 29	13	< 16	+ 2
15	64	- 68	14	92	+ 98	14	55	+ 52
16	< 16	+ 17	15	31	- 31	15	< 17	- 1
17	65	- 65	16	26	- 26	16	35	- 29
18	25	- 23	17	39	- 38	17	< 17	- 5
19	37	+ 42	18	34	- 34	18	46	- 46
20	< 17	- 4	19	< 17	+ 22	19	< 16	- 7
21	46	+ 56	20	< 17	+ 1	20	44	- 52
22	< 16	+ 4	21	26	+ 27	21	< 15	- 2
23	< 15	+ 12	22	< 15	+ 18	22	< 13	+ 11
24	19	+ 26	23	< 17	- 15	23	< 12	- 3
25	< 12	- 10	24	31	+ 33	24	35	+ 45
26	< 9	- 1	25	12	- 17			
	$0k2$			$0k4$			$0k6$	
0	82*	+ 92	0	40	+ 36	0	44	- 51
1	21	+ 26	1	49	+ 61	1	56	+ 58
2	19	+ 18	2	< 9	+ 16	2	< 12	- 16
3	64	+ 76	3	148*	+ 182	3	135	+ 153
4	89*	- 99	4	102*	- 109	4	< 13	+ 9
5	46	+ 42	5	64	+ 61	5	< 13	+ 12
6	153*	- 181	6	30	- 32	6	34	+ 33
7	102*	- 108	7	97	- 97	7	83	- 83
8	< 10	- 2	8	62	+ 70	8	24	- 22
9	57	- 61	9	33	- 31	9	55	- 44
						10	41	- 38

k	F_o	F_c	k	F_o	F_c	k	F_o	F_c
11	< 16	+ 6	19	< 13	0	11	34	- 28
12	< 17	+ 6	20	35	- 38	12	< 15	+ 5
13	75	+ 73	21	< 10	+ 10	13	< 14	+ 12
14	< 17	- 7				14	< 13	- 10
15	< 17	0		0k9		15	28	+ 24
16	22	+ 22	1	63	- 51	16	< 10	- 2
17	45	- 43	2	< 16	+ 5			
18	< 16	+ 11	3	< 16	+ 11		0,k,12	
19	40	- 49	4	23	+ 19	0	40	- 42
20	< 15	- 2	5	62	+ 60	1	< 16	- 12
21	17	- 27	6	54	- 49	2	< 16	- 10
22	< 12	- 6	7	67	+ 67	3	39	- 33
23	29	+ 34	8	34	- 32	4	26	+ 24
			9	39	- 37	5	20	+ 15
	0k7		10	< 17	- 9	6	44	+ 38
1	50	- 50	11	70	- 74	7	39	+ 34
2	56	+ 61	12	< 17	- 4	8	< 14	+ 5
3	51	+ 52	13	< 16	+ 14	9	< 14	+ 15
4	87	+ 93	14	< 16	+ 16	10	48	- 41
5	63	+ 63	15	39	+ 42	11	< 13	+ 5
6	54	- 45	16	< 15	+ 4	12	27	- 18
7	< 15	+ 8	17	34	+ 32	13	29	- 25
8	48	- 51	18	< 13	- 5	14	< 10	+ 13
9	66	- 69	19	< 11	- 23			
10	33	- 31	20	< 10	- 4		0,k,13	
11	54	- 51				1	14	- 14
12	53	+ 52		0,k,10		2	32	- 24
13	19	- 22	0	128	- 128	3	< 14	+ 10
14	72	+ 75	1	< 17	+ 16	4	33	- 27
15	< 17	- 6	2	39	- 30	5	< 13	+ 13
16	< 17	- 20	3	< 17	+ 4	6	34	+ 33
17	30	+ 25	4	50	+ 46	7	29	+ 26
18	30	- 33	5	29	- 31	8	36	+ 33
19	< 15	- 6	6	42	+ 38	9	27	- 16
20	16	- 14	7	< 17	- 20	10	14	+ 13
21	16	- 17	8	< 17	+ 10	11	38	- 33
22	13	+ 16	9	< 17	+ 10			
			10	59	- 58		0,k,14	
	0k8		11	< 16	+ 4	0	< 10	- 6
0	55	- 55	12	< 16	- 7	1	< 11	+ 2
1	< 15	- 11	13	< 15	- 18	2	< 10	+ 10
2	< 15	- 3	14	33	+ 31	3	41	- 36
3	53	+ 48	15	< 14	+ 6	4	19	+ 15
4	54	+ 54	16	36	+ 32	5	25	- 17
5	< 15	+ 4	17	< 12	+ 11	6	12	+ 11
6	67	+ 70	18	26	+ 28			
7	66	- 62					hk0 zone	
8	< 16	+ 13		0,k,11			0k0	
9	51	- 48	1	81	- 78	2	39	+ 46
10	68	- 72	2	< 17	- 16	4	102	- 129
11	< 17	- 2	3	32	+ 24	6	36	+ 19
12	38	- 39	4	< 17	- 14	8	< 14	- 1
13	60	+ 64	5	52	+ 45	10	130	+ 134
14	< 17	+ 15	6	56	+ 45	12	32	- 40
15	< 16	+ 16	7	23	+ 23	14	50	- 49
16	41	+ 43	8	20	+ 17	16	85	- 74
17	< 15	+ 15	9	27	- 25	18	82	- 95
18	< 14	+ 7	10	< 15	+ 3	20	62	+ 48

<i>k</i>	<i>F</i> _o	<i>F</i> _c	<i>k</i>	<i>F</i> _o	<i>F</i> _c	<i>k</i>	<i>F</i> _o	<i>F</i> _c
22	53	+ 66	21	< 21	+ 13	21	< 14	- 4
24	< 18	- 7	22	< 20	+ 1	22	21	- 20
26	34	- 34	23	33	+ 24			
			24	15	+ 7			
			25	28	+ 28			
	1 <i>k</i> 0						5 <i>k</i> 0	
0	189	+ 187				0	108	- 101
1	115	+ 110		3 <i>k</i> 0		1	67	- 56
2	73	+ 79	0	97	- 101	2	27	- 31
3	153	+ 147	1	36	+ 42	3	53	- 52
4	66	- 63	2	< 16	- 5	4	51	+ 49
5	48	- 42	3	46	+ 50	5	45	- 39
6	77	- 89	4	76	+ 80	6	40	+ 38
7	90	- 108	5	22	+ 25	7	< 23	+ 22
8	16	- 19	6	77	+ 76	8	< 23	- 22
9	66	- 69	7	84	- 81	9	< 23	+ 54
10	57	+ 60	8	< 19	+ 10	10	< 23	- 18
11	54	+ 51	9	68	- 75	11	44	- 47
12	18	+ 28	10	98	- 97	12	< 22	+ 4
13	62	+ 55	11	< 21	+ 26	13	< 22	- 40
14	30	- 38	12	33	- 41	14	< 20	+ 12
15	39	+ 42	13	58	+ 67	15	< 19	- 12
16	80	- 84	14	< 23	+ 19	16	34	+ 33
17	< 23	+ 24	15	43	+ 57	17	23	+ 21
18	23	- 26	16	47	+ 44	18	14	+ 19
19	57	- 53	17	23	- 22	19	37	+ 35
20	55	+ 54	18	< 23	+ 16			
21	< 22	+ 7	19	22	- 15		6 <i>k</i> 0	
22	29	+ 27	20	44	- 43	0	< 21	+ 9
23	33	+ 40	21	< 19	+ 22	1	47	- 43
24	< 17	+ 5	22	< 17	- 4	2	< 22	- 13
25	27	+ 33	23	35	+ 24	3	80	- 72
26	23	- 16	24	< 11	+ 1	4	< 22	- 3
						5	< 22	- 8
	2 <i>k</i> 0			4 <i>k</i> 0		6	< 21	+ 3
0	42	- 45	0	181	- 163	7	45	+ 45
1	104	+ 100	1	< 20	+ 12	8	< 21	- 7
2	50	+ 44	2	44	- 39	9	33	+ 30
3	144	+ 142	3	< 20	+ 4	10	21	+ 22
4	< 14	- 10	4	40	+ 37	11	19	- 20
5	37	+ 43	5	86	- 81	12	< 17	+ 7
6	< 15	- 9	6	53	+ 48	13	36	- 32
7	148	- 137	7	41	- 34	14	< 14	+ 4
8	< 16	0	8	35	+ 37	15	19	- 18
9	99	- 100	9	< 23	+ 17			
10	< 18	- 20	10	103	- 109		7 <i>k</i> 0	
11	71	+ 70	11	23	- 28	0	53	+ 53
12	< 20	+ 3	12	39	- 46	1	< 16	- 12
13	81	+ 81	13	< 23	- 15	2	< 16	- 12
14	< 22	+ 27	14	48	+ 52	3	39	- 39
15	63	+ 63	15	< 23	+ 3	4	40	- 38
16	< 23	+ 4	16	48	+ 53	5	< 15	- 6
17	32	- 32	17	25	+ 35	6	30	- 24
18	< 23	+ 10	18	27	+ 33	7	25	+ 26
19	59	- 55	19	< 18	+ 15	8	< 12	+ 4
20	< 22	+ 2	20	40	- 44	9	< 11	+ 5

Table 3. Dimensions of the coordination group.

$\left. \begin{array}{l} \text{Te}-\text{S}_1 = 2.532 \\ \text{Te}-\text{S}_2 = 2.469 \\ \text{Te}-\text{S}_3 = 2.857 \\ \text{Te}-\text{S}_3' = 3.016 \end{array} \right\} \pm 0.015 \text{ \AA}$	$\left. \begin{array}{l} \angle \text{S}_1-\text{Te}-\text{S}_2 = 95.4 \\ \angle \text{S}_2-\text{Te}-\text{S}_3 = 89.8 \\ \angle \text{S}_3-\text{Te}-\text{S}_3' = 82.8 \\ \angle \text{S}_3'-\text{Te}-\text{S}_1 = 92.1 \end{array} \right\} \pm 0.4^\circ$
$\left. \begin{array}{l} \text{S}_1-\text{C}_1 = 1.73 \\ \text{S}_2-\text{C}_2 = 1.77 \\ \text{S}_3-\text{C}_3 = 1.75 \end{array} \right\} \pm 0.07 \text{ \AA}$	$\left. \begin{array}{l} \angle \text{Te}-\text{S}_1-\text{C}_1 = 107 \\ \angle \text{Te}-\text{S}_2-\text{C}_2 = 104 \\ \angle \text{Te}-\text{S}_3-\text{C}_3 = 106 \end{array} \right\} \pm 2^\circ$

$B = 2.5 + 0.5 \cos^2\varphi$ in the $0kl$ zone and $2.0 + 0.4 \cos^2\varphi$ in the $hk0$ zone, where φ is the angle between the normal of the reflecting plane and the direction of maximum vibration of the atom, the b axis. The reliability index, R , with all observed reflections included, and with non-observed reflections included when $|F_c|$ exceeds the observable limit, is 0.106 for the $0kl$ data and 0.094 for the $hk0$ data.

The standard deviations of the atomic coordinates were estimated from the root-mean-square gradients of the difference maps and the curvatures of peaks in the electron density maps. Where overlapping occurred, which was the case particularly in the c -axis projection, the values were increased relative to those which would otherwise have applied. The resulting s.d. of bond lengths and angles involving the heavier atoms are included in Table 3. The s.d. of the carbon and nitrogen coordinates are probably 0.04–0.08 Å, and of the fluorine coordinates, 0.02–0.04 Å.

THE TELLURIUM(II)-THIOUREA CATION

Bond lengths and angles involving the tellurium and sulphur atoms, from the atomic coordinates of Table 1, are listed in Table 3, together with the estimated standard deviations.

The tellurium atom lies 2.20 Å from the symmetry centre, 0,0,0, and is surrounded by four thiourea sulphur atoms in a planar, distorted square arrangement, at Te—S distances of 2.47 to 3.02 Å and S—Te—S angles of 82.8° to 95.4°. Two of these sulphur atoms, S_3 and S_3' , are related to each other through the symmetry centre, and a centrosymmetric, planar arrangement of two tellurium atoms and six sulphur atoms results. The distances of the atoms from the least-squares plane through tellurium and the three sulphur atoms S_1 , S_2 , and S_3 , and through the corresponding atoms on the other side of the symmetry centre, and thus passing through the centre, are 0.020 Å for tellurium and -0.019 , 0.008 , and -0.025 Å, respectively, for S_1 , S_2 and S_3 . For sulphur the deviations are within the limits of error. A view of the cation is shown in Fig. 2.

The sulphur atoms S_3 and S_3' , which form the bridges of the binuclear complex, are *trans* to S_1 and S_2 , respectively, at $\text{S}_1-\text{Te}-\text{S}_3$ and $\text{S}_2-\text{Te}-\text{S}_3'$ angles of 174.6° and 172.6°. The bridging Te—S bonds, $\text{Te}-\text{S}_3 = 2.86$ Å and $\text{Te}-\text{S}_3' = 3.02$ Å, are longer than the other two, $\text{Te}-\text{S}_1 = 2.53$ Å and $\text{Te}-\text{S}_2 = 2.47$ Å. However, the mean length of *trans*-positioned Te—S

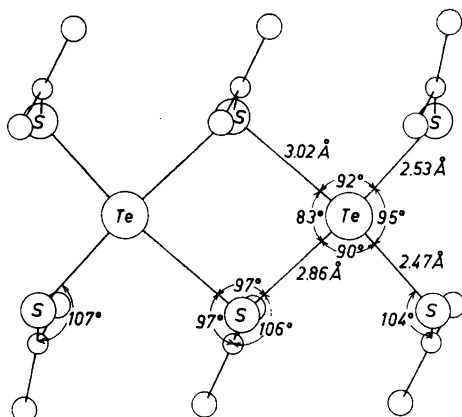


Fig. 2. The di- μ -thiourea-bis(dithiourea-tellurium(II)) cation, as seen normal to the least-squares plane of the tellurium and sulphur atoms.

bonds, 2.70 Å for Te-S₁ and Te-S₃ and 2.75 Å for Te-S₂ and Te-S_{3'}, is only in the case of the latter, more asymmetric pair significantly larger than the length of the Te-S bond in the centrosymmetric, square-planar Te(tu)₄²⁺ cation,⁴ 2.68–2.69 Å.

The carbon and nitrogen coordinates give C–N bond lengths of 1.31 to 1.38 Å, S–C–N angles 115°–124° and N–C–N angles 118°–123°, with rather large uncertainties. The thiourea groups are planar within the error, the distances of the middle atom, carbon, from the least-squares planes of the groups, with sulphur given three times the weight of carbon and nitrogen, are less than 0.05 Å. The six thiourea groups of a binuclear cation are nearly parallel, the least-squares planes of the three non-equivalent groups making angles of 4° to 12° with each other, and angles of 79° to 83° with the least-squares plane of the tellurium and sulphur atoms.

THE PACKING IN THE CRYSTAL

In the hydrogendifluoride ions, F₁–F₂ = 2.28 Å and F₃–F₄ = 2.30 Å, with standard deviations of about 0.04 Å. For sodium⁹ and potassium¹⁰ hydrogendifluoride, the reported F–F distances are 2.264 ± 0.003 Å and 2.277 ± 0.006 Å, respectively, and for ammonium hydrogendifluoride,¹¹ 2.269 and 2.275 Å, each ± 0.005 Å.

The cations and the hydrogendifluoride anions are in the crystals held together through N–H...F hydrogen bonds, extending from the thiourea amino groups. The lengths of these hydrogen bonds are listed in Table 4, together with the respective C–N...F angles, and the distances of the fluoride ions from the least-squares plane of the thiourea group. The coordinates, for fluorine, refer to x, y, z as listed in Table 1. The standard deviation of the N–H...F lengths is about 0.07 Å; the average length, 2.82 Å, agrees well with the N–H...F distances in ammonium hydrogendifluoride,¹¹ 2.797 and 2.822 Å, each ± 0.005 Å. The average C–N...F angle is 118°.

Table 4. Nitrogen-fluorine distances.

Distance	Length N...F	Angle C-N...F	Distance from plane
$\text{N}_1 \cdots \text{F}_3$ ($x-1, y, 1+z$)	2.63 Å	124°	0.41 Å
$\text{N}_2 \cdots \text{F}_2$ ($x, \frac{1}{2}-y, \frac{1}{2}+z$)	2.91	124	0.96
$\text{N}_2 \cdots \text{F}_3$ ($x, y, 1+z$)	2.89	110	-0.54
$\text{N}_3 \cdots \text{F}_1$ ($x-1, y, z$)	2.79	104	0.39
$\text{N}_3 \cdots \text{F}_2$ ($x-1, \frac{1}{2}-y, \frac{1}{2}+z$)	2.88	123	0.45
$\text{N}_4 \cdots \text{F}_1$ (x, y, z)	2.92	125	-0.12
$\text{N}_5 \cdots \text{F}_4$ ($x-1, y, z$)	2.79	111	0.84
$\text{N}_6 \cdots \text{F}_4$ (x, y, z)	2.75	121	-0.05

Each fluoride ion is engaged in two N—H...F bonds, which makes a total of eight in the asymmetric unit. The nitrogen atoms N_2 and N_3 form two each, at F...N...F angles of 124° and 128°, respectively, the remaining four nitrogen atoms form one each, all eight to different fluoride ions.

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