

## The Crystal and Molecular Structure of a *trans* Square-Planar Complex of Tellurium Dimethanethiosulphonate with Thiourea

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The complex,  $\text{Te}(\text{tu})_2(\text{S}_2\text{O}_2\text{CH}_3)_2$  where tu = thiourea, crystallizes in the space group  $C_{2h}^2 - P_2^1/n$  with two molecules per unit cell, and  $a = 12.50 \text{ \AA}$ ,  $b = 5.60 \text{ \AA}$ ,  $c = 12.80 \text{ \AA}$ ,  $\beta = 98^\circ$ . The crystal and molecular structure has been determined by X-ray methods, and refined by least squares for the three principal zones.

The tellurium atoms lie in centres of symmetry, and are each bonded to two thiourea sulphur atoms and two thiosulphonate sulphur atoms in a *trans* square-planar arrangement. The  $\text{TeS}_4$  group has the dimensions,  $\text{Te}-\text{S}(\text{thiourea}) = 2.67 \text{ \AA}$ ,  $\text{Te}-\text{S}(\text{thiosulphonate}) = 2.68 \text{ \AA}$ , each  $\pm 0.015 \text{ \AA}$ ,  $\angle \text{S}-\text{Te}-\text{S} = 90.6 \pm 0.5^\circ$ . The  $\text{Te}-\text{S}$  (thiosulphonate) bond is about  $0.30 \text{ \AA}$  longer than in uncomplexed tellurium dimethanethiosulphonate.

The methanethiosulphonate S-S bond in the complex is found to be  $2.02 \pm 0.02 \text{ \AA}$ , as compared with  $1.98 \pm 0.01 \text{ \AA}$  in ionic sodium methanethiosulphonate monohydrate, and  $2.10-2.12 \text{ \AA}$  in covalent, uncomplexed methanethiosulphonates.

Tellurium dimethanethiosulphonate,  $\text{Te}(\text{S}_2\text{O}_2\text{CH}_3)_2$ , was prepared in 1950 by one of us,<sup>1</sup> and its crystal structure determined.<sup>2</sup> Nucleophilic reagents, like thiosulphate, xanthate, and dithiocarbamate ions, attack tellurium to release the methanethiosulphonate groups as ions, and forming telluropentathionate, tellurium xanthates, and dithiocarbamates.<sup>1,3</sup> It was found recently<sup>4</sup> that the nucleophile, thiourea, may not effect a substitution but adds to tellurium to give a square-planar tellurium(II) complex. Its crystal and molecular structure is reported here.

### CRYSTAL DATA

The crystals of dimethanethiosulphonatodithioureatellurium(II),  $\text{Te}(\text{tu})_2(\text{S}_2\text{O}_2\text{CH}_3)_2$ , occur as yellow, monoclinic prisms extended along the *b* axis, with <sup>4</sup>  $a = 12.50 \text{ \AA}$ ,  $b = 5.60 \text{ \AA}$ ,  $c = 12.80 \text{ \AA}$ ,  $\beta = 98^\circ$ , and two molecules per

unit cell. The space group, from systematic absences, is  $C_{2h}^5 - P2_1/n$ , which requires that the tellurium atoms lie in centres of symmetry.

Intensities were estimated visually from zero-layer Weissenberg photographs around the  $a$ ,  $b$ , and  $c$  axes, using  $CuK\alpha$  radiation and crystals with cross-sections approximately  $0.1 \times 0.1$  mm ( $\mu = 207$  cm $^{-1}$ ). The  $b$ - and  $c$ -axis photographs were non-integrated and taken with a double-film technique, the  $a$ -axis photographs were integrated, multiple-film. 64  $0kl$ , 179  $h0l$ , and 67  $hk0$  reflections were observed, out of 89, 199, and 88, respectively, accessible with  $CuK\alpha$  radiation. No corrections for absorption or extinction were made.

#### THE STRUCTURE ANALYSIS

The  $b$ -axis projection was solved in a straight-forward way through a Fourier synthesis of the  $h0l$  reflections with positive signs. Five of these later turned out to be negative. In the  $c$ -axis projection, where tellurium does not contribute to  $hk0$  reflections with  $h + k$  odd, a Fourier synthesis was made of the strongest  $h + k$  even reflections with positive signs. In the resulting map, which had false symmetry due to the omission of the  $h + k$  odd reflections, the correct set of sulphur positions could be picked out by dimensional considerations. The  $b$ - and  $c$ -axis projections were refined through Fourier syntheses and difference syntheses, to  $R$  indexes of about 0.11 for both zones. The  $h0l$  Fourier map is shown in Fig. 1.

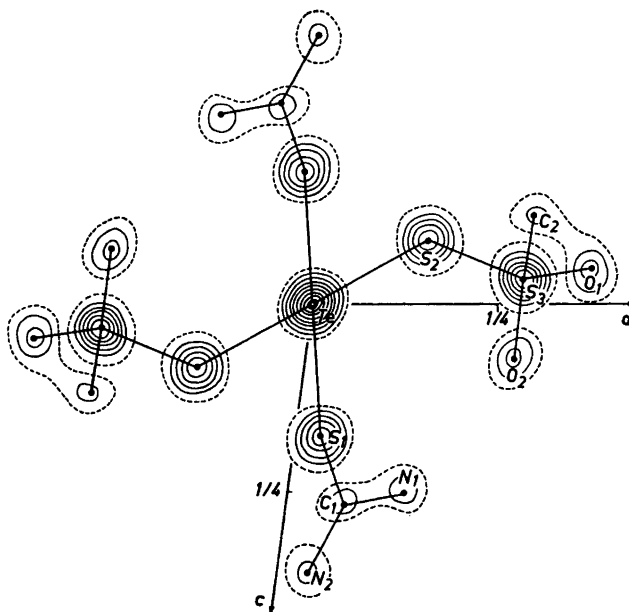


Fig. 1. Electron density projection of dimethanethiosulphonatodithioureate tellurium(II) along the  $b$  axis. The 4-electron line is dashed. Contour intervals:  $13$  e. $\text{\AA}^{-3}$  for tellurium,  $4$  e. $\text{\AA}^{-3}$  for sulphur, and  $3$  e. $\text{\AA}^{-3}$  for the other atoms. The strong reflections  $002$ ,  $10\bar{1}$ , and  $30\bar{1}$  are included in the map with their calculated instead of observed values.

Three cycles of least-squares refinement for the  $0kl$ ,  $h0l$ , and  $hk0$  data were carried out on the IBM 650 computer, using the modified LS II program.<sup>5</sup> This program is limited to isotropic temperature factors, and anisotropy for tellurium was simulated by Kartha and Ahmed's method,<sup>6</sup> by placing one half tellurium atom in general position a little outside the symmetry centre. The reflections were assigned equal weight. In the last cycle, six low order reflections, 002, 011,  $10\bar{1}$ ,  $30\bar{1}$ , 110, and 310, which had markedly higher calculated than observed values, were excluded, and likewise, one of the two sets of axial reflections contained in the three sets of zonal data. The last cycle thus comprised about 290 independent non-zero reflections. For the lighter atoms, carbon, nitrogen, and oxygen of the thiourea and methanethiosulphonate groups, coordinate shifts were in some cases unreasonable and were not considered to be genuine, the coordinates were instead adjusted on the basis of the known dimensions of the groups in thiourea,<sup>7</sup> dichlorobis(thiourea)-zinc,<sup>8</sup> and sodium methanethiosulphonate monohydrate.<sup>9</sup>

Table 1. Atomic coordinates for dimethanethiosulphonatodithioureatellurium(II), in fractions of monoclinic cell edges, and values of temperature parameters  $B$ , in  $\text{\AA}^2$  units. Origin at a centre of symmetry.

	$x$	$y$	$z$	$B$
Te	0	0	0	1.45*
S <sub>1</sub>	0.0355	0.2543	0.1776	3.42
C <sub>1</sub>	0.0840	0.0312	0.2685	2.88
N <sub>1</sub>	0.1602	-0.1208	0.2478	4.26
N <sub>2</sub>	0.0440	0.0225	0.3595	4.49
S <sub>2</sub>	0.1459	0.2618	-0.0848	3.50
S <sub>3</sub>	0.2837	0.0821	-0.0337	2.57
C <sub>2</sub>	0.2910	-0.1620	-0.1225	5.21
O <sub>1</sub>	0.3740	0.2430	-0.0465	3.76
O <sub>2</sub>	0.2879	-0.0024	0.0730	4.59

The final atomic coordinates are listed in Table 1, together with the values of  $B$  in the temperature factor  $\exp[-B(\sin^2\theta/\lambda^2)]$ . The structure factors calculated from these coordinates are listed in Table 2 together with the observed ones. They are based on the scattering curve of Thomas and Umeda<sup>10</sup> for tellurium, that of Tomiie and Stam<sup>11</sup> for sulphur, and those of Berghuis *et al.*<sup>12</sup> for oxygen, nitrogen and carbon. The overall reliability index,  $R$ , with non-observed reflections included when  $|F_c|$  exceeds the observable limit, and with the six low order reflections referred to above, not included, is 0.098.

The approximate standard deviations of the atomic coordinates, from the output of the last least-squares cycle, are 0.01, 0.02, 0.01  $\text{\AA}$  for sulphur, 0.03, 0.07, 0.03  $\text{\AA}$  for oxygen, 0.04, 0.08, 0.04  $\text{\AA}$  for nitrogen, and 0.05, 0.09, 0.05  $\text{\AA}$  for carbon, in the  $x$ ,  $y$ , and  $z$  directions, respectively.

\* The final coordinates for one half tellurium, used to simulate the anisotropy of the atom, were  $x = -0.0059$ ,  $y = 0.0178$ ,  $z = 0.0081$ .

Table 2. Observed and calculated  $hk0$ ,  $h0l$  and  $0kl$  structure factors for dimethanethio-sulphonatodithioureate tellurium(II).

$h$	$F_o$	$F_c$	$h$	$F_o$	$F_c$	$h$	$F_o$	$F_c$
	$h00$		12	11	- 6		$h0\bar{1}$	
2	48	+ 59	13	20	+ 25	1	77	+ 103
4	83	+ 91	14	< 7	+ 10	3	144	+ 187
6	88	+ 90		$h40$		5	83	+ 89
8	61	+ 57	1	34	- 29	7	151	+ 163
10	32	+ 30	2	83	+ 77	9	15	+ 15
12	< 13	+ 6	3	< 12	+ 12	11	52	+ 52
14	42	+ 41	4	33	+ 34	13	45	+ 43
	$h10$		5	15	- 7	15	16	+ 16
1	107	+ 120	6	54	+ 50		$h02$	
2	38	- 34	7	< 13	+ 3	2	19	+ 12
3	115	+ 134	8	37	+ 41	4	65	+ 65
4	35	- 30	9	< 13	+ 8	6	36	+ 30
5	23	+ 21	10	< 12	+ 14	8	83	+ 84
6	17	- 16	11	< 11	0	10	48	+ 42
7	96	+ 94	12	14	+ 17	12	< 12	+ 10
8	48	- 44	13	< 6	+ 14	14	44	+ 47
9	16	+ 17		$h50$			$h0\bar{2}$	
10	< 13	- 4	1	34	+ 37	2	30	- 28
11	52	+ 55	2	22	- 14	4	86	+ 95
12	< 14	+ 3	3	36	+ 39	6	91	+ 99
13	27	+ 29	4	17	- 12	8	64	+ 62
14	< 10	- 1	5	38	+ 39	10	75	+ 73
15	16	+ 21	6	< 13	+ 5	12	29	+ 32
	$h20$		7	13	+ 14	14	44	+ 44
1	10	- 9	8	22	- 17		$h03$	
2	44	+ 39	9	31	+ 33	1	130	+ 144
3	18	+ 18	10	< 9	+ 7	3	104	+ 118
4	110	+ 112	11	14	+ 19	5	15	+ 2
5	33	- 25		$h60$		7	73	+ 70
6	22	+ 23	1	< 11	0	9	57	+ 53
7	13	+ 13	2	24	+ 30	11	56	+ 57
8	25	+ 28	3	< 11	+ 1	13	25	+ 28
9	15	+ 13	4	19	+ 24		$h0\bar{3}$	
10	68	+ 71	5	< 10	+ 1	1	65	- 57
11	16	- 7	6	12	+ 18	3	62	+ 57
12	35	+ 39	7	< 9	+ 6	5	91	+ 87
13	< 11	+ 11	8	15	+ 17	7	69	+ 70
14	25	+ 32		$h70$		9	< 11	+ 14
15	< 6	- 3	1	18	+ 26	11	69	+ 68
	$h30$		2	< 7	+ 3	13	43	+ 46
1	85	+ 82		$h01$		15	9	+ 13
2	45	+ 40	1	74	+ 91		$h04$	
3	68	+ 70	3	68	+ 75	2	73	+ 72
4	27	- 20	5	26	- 16	4	80	+ 78
5	48	+ 45	7	90	+ 91	6	60	+ 55
6	32	+ 37	9	< 11	+ 4	8	95	+ 96
7	41	+ 47	11	49	+ 42	10	68	+ 60
8	17	+ 20	13	23	+ 23	12	38	+ 38
9	35	+ 36	15	22	+ 26	14	29	+ 34
10	23	+ 27						
11	25	+ 30						

$h$	$F_o$	$F_c$	$h$	$F_o$	$F_c$	$h$	$F_o$	$F_c$
	$h0\bar{4}$		7	54	+ 53	5	54	+ 57
2	37	+ 31	9	78	+ 74	7	31	+ 35
4	56	+ 54	11	36	+ 36	9	18	+ 25
6	46	+ 39	13	31	+ 27			
8	< 11	+ 1	15	< 6	+ 7		$h,0,\bar{11}$	
10	60	+ 55				1	53	+ 56
12	38	+ 34		$h08$		3	49	+ 40
14	26	+ 28	2	< 11	+ 3	5	29	+ 25
			4	63	+ 67	7	24	+ 22
	$h05$		6	31	+ 29	9	34	+ 31
1	112	+ 114	8	43	+ 40	11	14	- 8
3	118	+ 136	10	23	+ 24			
5	94	+ 96	12	39	+ 49		$h,0,12$	
7	58	+ 52				2	36	+ 38
9	64	+ 62		$h0\bar{8}$		4	18	+ 18
11	54	+ 54	2	97	+ 96	6	18	+ 25
13	< 9	+ 5	4	19	+ 21	8	24	+ 32
			6	54	+ 48			
	$h0\bar{5}$		8	24	+ 22		$h,0,\bar{12}$	
1	63	+ 50	10	59	+ 60	2	59	+ 58
3	101	+ 105	12	24	+ 23	4	23	+ 22
5	66	+ 64	14	19	+ 23	6	66	+ 65
7	29	+ 25				8	20	+ 19
9	17	+ 9		$h09$		10	< 9	+ 10
11	22	+ 21	1	50	+ 49	12	20	+ 19
13	28	+ 22	3	< 12	0			
15	10	+ 7	5	77	+ 76		$h,0,13$	
			7	42	+ 41	1	33	+ 37
	$h06$		9	31	+ 34	3	< 11	+ 5
2	86	+ 83	11	36	+ 46	5	23	+ 29
4	108	+ 115				7	16	+ 20
6	26	+ 23		$h0\bar{9}$				
8	41	+ 38	1	30	+ 26		$h,0,\bar{13}$	
10	24	+ 23	3	59	+ 51	1	25	+ 26
12	23	+ 25	5	< 12	+ 4	3	19	+ 14
			7	44	+ 38	5	37	+ 39
	$h0\bar{6}$		9	62	+ 53	7	27	+ 29
2	138	+ 146	11	15	- 4	9	30	+ 29
4	113	+ 118	13	27	+ 29	11	< 6	+ 6
6	63	+ 58					$h,0,14$	
8	33	+ 33		$h,0,10$		2	33	+ 38
10	72	+ 67	2	42	+ 38	4	14	+ 12
12	< 12	+ 5	4	75	+ 77			
14	< 9	+ 5	6	48	+ 49		$h,0,\bar{14}$	
			8	50	+ 50	2	19	+ 15
	$h07$		10	16	+ 16	4	14	+ 13
1	78	+ 76				6	27	+ 28
3	54	+ 42		$h,0,\bar{10}$		8	15	+ 14
5	51	+ 46	2	66	+ 66			
7	< 12	+ 11	4	20	- 14		$h,0,15$	
9	26	+ 28	6	46	+ 37	1	20	+ 25
11	41	+ 44	8	16	+ 11	3	17	+ 20
13	15	+ 16	10	35	+ 24			
			12	11	+ 7		$h,0,\bar{15}$	
	$h0\bar{7}$					1	14	+ 15
1	30	+ 23		$h,0,11$		3	< 9	+ 6
3	88	+ 97	1	94	+ 94	5	24	+ 24
5	58	+ 57	3	25	+ 18	7	< 7	- 4

<i>h</i>	<i>F</i> <sub>o</sub>	<i>F</i> <sub>c</sub>	<i>l</i>	<i>F</i> <sub>o</sub>	<i>F</i> <sub>c</sub>	<i>l</i>	<i>F</i> <sub>o</sub>	<i>F</i> <sub>c</sub>
	<i>h,0,16</i>							
2	16	+ 18	4	103	+ 100	6	35	+ 35
4	15	+ 17	5	41	+ 34	7	< 12	+ 4
			6	59	+ 57	8	< 12	+ 11
<i>l</i>	<i>00l</i>		7	29	+ 20	9	< 12	0
2	108	+ 136	8	81	+ 80	10	34	+ 34
4	80	+ 84	9	17	+ 15	11	< 10	0
6	47	+ 46	10	18	+ 22	12	30	+ 38
8	30	+ 28	11	< 12	+ 2	13	< 7	- 3
10	33	+ 29	12	12	+ 11			
12	44	+ 42	13	< 11	+ 2		<i>05l</i>	
14	< 11	+ 9	14	25	+ 27	1	17	+ 21
16	< 6	+ 11	15	< 7	- 7	2	< 12	+ 10
						3	29	+ 30
				<i>03l</i>		4	29	+ 28
1	<i>01l</i>		1	70	+ 69	5	18	+ 26
2	130	+ 155	2	9	+ 6	6	< 12	- 2
3	21	+ 18	3	73	+ 74	7	28	+ 28
4	133	+ 144	4	47	- 42	8	12	- 12
5	106	+ 107	5	52	+ 48	9	26	+ 26
6	76	+ 75	6	29	+ 20	10	9	+ 3
7	24	+ 22	7	33	+ 33	11	18	+ 24
8	52	+ 51	8	38	+ 31			
9	< 10	- 16	9	24	+ 30		<i>06l</i>	
10	38	+ 36	10	< 12	+ 6	0	< 12	+ 1
11	16	+ 9	11	37	+ 44	1	< 11	- 7
12	37	+ 33	12	< 11	+ 4	2	14	+ 16
13	< 12	- 10	13	21	+ 24	3	< 11	- 2
14	12	+ 15	14	< 8	- 7	4	20	+ 21
15	< 10	+ 5				5	10	- 9
	10	+ 15		<i>04l</i>		6	14	+ 19
			0	97	+ 98	7	< 8	0
			1	< 10	0	8	22	+ 32
	<i>02l</i>		2	47	+ 48			
0	37	+ 43	3	21	+ 18		<i>07l</i>	
1	< 7	+ 6	4	23	+ 22	1	16	+ 15
2	83	+ 82	5	19	+ 11	2	< 6	- 3
3	10	- 6						

## RESULTS

Bond lengths and angles involving the TeS<sub>4</sub> coordination group, as calculated from the atomic coordinates of Table 1, are listed in Table 3 together

Table 3. Dimensions of the coordination group.

Te-S <sub>1</sub> = 2.667 ± 0.015 Å	Te-S <sub>2</sub> = 2.684 ± 0.015 Å
∠S <sub>1</sub> -Te-S <sub>2</sub> = 90.6 ± 0.5°	
S <sub>1</sub> -C <sub>1</sub> = 1.76 ± 0.06 Å	∠Te-S <sub>1</sub> -C <sub>1</sub> = 100.7 ± 2.1°
S <sub>2</sub> -S <sub>3</sub> = 2.024 ± 0.018	∠Te-S <sub>2</sub> -S <sub>3</sub> = 101.2 ± 0.7

with the standard deviations. With tellurium in a centre of symmetry, the TeS<sub>4</sub> coordination group is exactly planar. In the tables, S<sub>1</sub> denotes the thiourea sulphur atom, and S<sub>2</sub> and S<sub>3</sub> the divalent and the sulphonate sulphur atom,

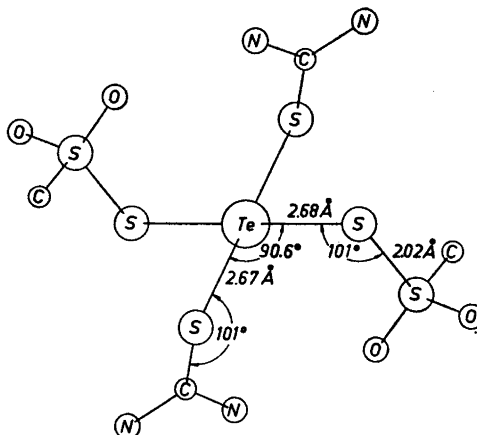


Fig. 2. The molecule as seen along the  $b$  axis.

respectively, of the methanethiosulphonate ligand. A drawing of the molecule is reproduced in Fig. 2.

The Te—S bond lengths are within the error the same as in the tetra-thioureate tellurium(II) cation,  $\text{Te}(\text{tu})_4^{2+}$ , and in *trans*-dibromo- and *trans*-diiodo-bis(ethylenethiourea)tellurium(II),  $\text{Te}(\text{etu})_2\text{Br}_2$  and  $\text{Te}(\text{etu})_2\text{I}_2$ . There, the found lengths<sup>13,14</sup> are 2.677 to 2.690 Å.

In uncomplexed tellurium dimethanethiosulphonate,<sup>2</sup> the Te—S bonds are 2.35 and 2.36 Å, each  $\pm 0.03$  Å, and the S—Te—S angle  $100 \pm 2^\circ$ . In tellurium dibenzenethiosulphonate,<sup>15</sup> Te—S =  $2.41 \pm 0.03$  Å,  $\angle \text{S—Te—S} = 97 \pm 2^\circ$ ; in tellurium di-*p*-toluenethiosulphonate,<sup>16</sup> Te—S =  $2.41 \pm 0.04$  Å,  $\angle \text{S—Te—S} = 96 \pm 3^\circ$ ; in ammonium telluropentathionate,<sup>17</sup> Te—S = 2.35 and 2.36 Å, each  $\pm 0.03$  Å,  $\angle \text{S—Te—S} = 103 \pm 2^\circ$ ; in barium telluropentathionate dihydrate,<sup>18</sup> Te—S =  $2.34 \pm 0.02$  Å,  $\angle \text{S—Te—S} = 101 \pm 1^\circ$ . The found Te—S bond lengths in uncomplexed compounds thus vary between 2.34 and 2.41 Å, and the S—Te—S angle, between  $96^\circ$  and  $103^\circ$ . The upper limit, 2.41 Å, is equal to the single covalent Te—S bond length. The effect of increasing the S—Te—S angle to  $180^\circ$ , through complexing with two thiourea molecules in *trans* positions, is thus to increase the Te—S bond length by about 0.30 Å.

The S—S bond length in the methanethiosulphonate group,  $2.024 \pm 0.018$  Å, is to be compared with the length,  $1.98 \pm 0.01$  Å, in ionic sodium methanethiosulphonate monohydrate,<sup>9</sup> and 2.10–2.12 Å in covalent, uncomplexed methanethiosulphonates. In tellurium dimethanethiosulphonate,<sup>2</sup> S—S = 2.13 and 2.16 Å, each  $\pm 0.03$  Å, but especially the latter value is probably too high; in the analogs referred to above, the values range from 2.08 to 2.12 Å with a weighted average of 2.10 Å; in dimethanesulphonyl disulphide,<sup>19</sup> S—S =  $2.10 \pm 0.02$  Å. The values reported for the S—S bond in the thiosulphate ion range from 1.96 to 2.02 Å, with a weighted average of 1.99 Å, *cf.* Ref. 9. In the covalently bonded thiosulphate groups of the tetra-, penta-, and hexathionate ions, and the seleno- and telluropentathionate ions, the bond is 2.10–2.12 Å.<sup>20</sup> A shorter methanethiosulphonate S—S bond in the complex, relative to covalent methanethiosulphonates, indicates for the Te—S bond a

covalency lower than one, and would be in accordance with a bonding scheme, based on tellurium  $5p$  orbitals, for tellurium(II) complexes.<sup>21</sup>

The rather inaccurate carbon, nitrogen, and oxygen coordinates correspond to C—N = 1.33 Å,  $\angle$ S—C—N = 121° and 118°,  $\angle$ N—C—N = 121° in the thiourea group, and S—O = 1.44 and 1.47 Å, S—C = 1.79 Å,  $\angle$ S—S—O = 107° and 113°,  $\angle$ S—S—C = 107°,  $\angle$ O—S—O = 112°,  $\angle$ C—S—O = 107° and 111° in the methanethiosulphonate group. The thiourea group is planar within 0.01 Å; its least-squares plane makes an angle of 79° with the TeS<sub>4</sub> plane.

The hydrogen atoms of the thiourea group appear to engage in hydrogen bonding to the thiosulphonate oxygen atoms. One such approach, N<sub>1</sub>...O<sub>2</sub> = 3.00 Å, at a C—N...O angle of 119°, occurs within the molecule. Intermolecular approaches are, N<sub>3</sub>...O<sub>2</sub>' = 3.14 Å, N<sub>2</sub>...O<sub>1</sub>' = 2.92 Å, N<sub>2</sub>...O<sub>1</sub> = 2.90 Å, at C—N...O angles of 112°, 127°, and 137°, respectively, where O<sub>2</sub>' and O<sub>1</sub>' are at  $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$  relative to O<sub>2</sub> and O<sub>1</sub>, respectively, and O<sub>1</sub>'' is at  $x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z$  relative to O<sub>1</sub>. These distances and angles are in the range found for N—H...O hydrogen bonds in other compounds.<sup>22,23</sup> All four hydrogen atoms of the thiourea group thus probably engage in hydrogen bonding, and each of the two thiosulphonate oxygen atoms engages in two hydrogen bonds. Those from N<sub>1</sub> to O<sub>2</sub> atoms occur around the screw axis  $\frac{1}{4}, y, \frac{1}{4}$ , and those from N<sub>2</sub> to O<sub>1</sub> atoms around the symmetry centre at  $0, 0, \frac{1}{2}$ .

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