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The Crystal and Molecular Structures of Two Forms of a *trans* Square-Planar Complex of Tellurium Dimethanethiosulphonate with Ethylenethiourea

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The complex, *trans*-dimethanethiosulphonatobis(ethylenethiourea)tellurium(II), $Te(etu)_2(S_2O_2CH_3)_2$, crystallizes in two forms: a triclinic one (I), space group $P\bar{1}$ (No. 2) with $Z=1$, $a=9.519(2)$ Å, $b=10.210(2)$ Å, $c=5.385(1)$ Å, $\alpha=98.88(2)°$, $\beta=105.54(2)°$, $\gamma=98.89(2)°$; and a monoclinic one (II), space group $P2_1/c$ (No. 14) with $Z=2$, $a=9.616(4)$ Å, $b=10.850(6)$ Å, $c=10.672(4)$ Å, $\beta=119.21(4)°$. In both forms, the tellurium atoms lie in centres of symmetry.

The crystal and molecular structures of both forms have been determined by three-dimensional X-ray methods, and refined by least squares procedures based on 912 independent, observed reflections for I and 800 for II.

The centrosymmetric, square-planar TeS_4 coordination groups are very like in the two dimorphs: Te–S(etu) = 2.663(6) Å in I and 2.687(5) Å in II, Te–S(thiosulphonate) = 2.694(6) Å in I and 2.685(4) Å in II, ∠ S–Te–S = 91.6(2)° in I and 92.6(2)° in II. The methanethiosulphonate S–S bond is 2.014(9) Å in I and 2.015(7) Å in II, ∠ Te–S–S = 103.9(3)° in I and 102.3(2)° in II. There are, from one dimorph to the other, some small differences in the rotational positions of the ligand groups.

Complexes of tellurium dimethanethiosulphonate,[1,2] $Te(S_2O_2CH_3)_2$, with thiourea and substituted thioureas as ligands were reported in 1961.[3,4] The crystal and molecular structure of the square-planar *trans* thiourea complex, $Te(tu)_2(S_2O_2CH_3)_2$, has been determined.[5] This paper reports the structures of two crystalline forms of the corresponding ethylenethiourea complex.

CRYSTAL DATA

The triclinic dimorph (I) of *trans*-dimethanethiosulphonatobis(ethylenethiourea)tellurium(II), $Te(etu)_2(S_2O_2CH_3)_2$, occurs as prisms extended along the *c* axis, bounded by {100} and {010} and terminated by {01$\bar{1}$}. This

dimorph was earlier,[3] due to twinning, erroneously described as *C*-centered monoclinic. It appears to be unstable, judging from extraneous reflections appearing on X-ray photographs of samples kept for a year in a refrigerator. The unit cell dimensions are, $a = 9.519(2)$ Å, $b = 10.210(2)$ Å, $c = 5.385(1)$ Å, $\alpha = 98.88(2)°$, $\beta = 105.54(2)°$, $\gamma = 98.89(2)°$. The space group is $P\bar{1}$ (No. 2) and there is one molecule per unit cell; density, calc. 1.89, found [3] 1.89 g/cm³. Cell volume, 487.6 Å³.

The monoclinic dimorph (II) occurs as plates {100} with edges along the *bc* diagonals, and with $a = 9.616(4)$ Å, $b = 10.850(6)$ Å, $c = 10.672(4)$ Å, $\beta = 119.21(4)°$. The space group is $P2_1/c$ (No. 14) and there are two molecules per unit cell; density, calc. 1.89, found [3] 1.89 g/cm³. Cell volume, 971.9 Å³, or 486.0 Å³ per molecule.

EXPERIMENTAL

The procedure used [3] for the preparation of the compound was modified slightly. 1.30 g of tetrakis(ethylenethiourea)tellurium(II) dichloride dihydrate [6] and 0.68 g (a slight excess) of sodium methanethiosulphonate monohydrate were dissolved in 10 ml of dimethylformamide at room temperature. The solution was filtered, and 10 ml of methanol was added to the filtrate under gentle swirling. On standing at room temperature, the product crystallized. After an hour or two, the crystals were filtered off, and washed with methanol, and then with ether. Yield, about 0.85 g, or 76 %.

The product consisted of a mixture of the two dimorphs. The crystals of the two forms could be readily distinguished and picked out under a microscope, on the basis of their different shapes. The colour is the same for both dimorphs, greenish-yellow.

Unit cell dimensions, as given above, were determined from high-order reflections on zero-layer Weissenberg photographs, $\lambda(CuK\alpha_1) = 1.5405$ Å, and evaluated by means of a least squares procedure, from 68 observed 2θ values for I and 34 for II.

Intensities were estimated visually from multiple-film, integrated, equi-inclination Weissenberg photographs taken with Ni-filtered $CuK\alpha$ radiation, except for the *hk*0 data of I which were estimated from a non-integrated film set. The following layers were photographed and used: 0*kl*, 1*kl*, 2*kl*, *hk*0, and *hk*1 for I, and *h*0*l*, *h*1*l*, *h*2*l*, *hk*0, *hk*1, and *hk*2 for II. This gave 1006 independent, accessible reflections for both dimorphs, of which 912 and 800, respectively, were observed with measurable intensities. The crystals used for the intensity photographs had cross-sections 0.08×0.07 mm for the *a*-axis photographs and 0.10×0.07 mm for the first-layer *c*-axis photographs of I, and 0.11×0.08 mm for the *b*-axis photographs and 0.10×0.09 mm for the *c*-axis photographs of II. In the case of the zero-layer *c*-axis photographs of I, a crystal with a slightly smaller cross-section than for the first-layer photographs was used. No corrections for absorption were made ($\mu = 186$ cm^{-1} for I and 187 cm^{-1} for II).

The calculated structure factors were based on the scattering curves listed in *International Tables* (Ref. 7, pp. 202, 211). The tellurium scattering curve, and in the case of II also the sulphur scattering curve, were corrected for anomalous dispersion using the $\Delta f'$ and $\Delta f''$ values given by Cromer,[8] by taking the amplitude of f as the corrected value.

Least squares refinement was in the case of I first carried out on an IBM 1620 computer using a program designed by Mair.[9] The refinement of II, and the final refinement of I, were carried out on an IBM 360/50 H computer with a full-matrix least squares program minimizing the function

$$r = \sum W(|F_o| - K|F_c|)^2$$

where K is a scale factor. The weight is defined by $W = 1/\sigma^2(F_o)$, where $\sigma(F_o)$ is the estimated standard deviation in F_o. Non-observed reflections for which $K|F_c|$ exceeds the observable limit, are included in the refinement with $|F_o|$ equal to the observable limit.

Most of the IBM 360/50 H 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 360/50 H computer by Dr. D. Rabinovich.

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

	x	y	z	U
Te	0	0	0	
S_1	0.2381(7)	0.1861(5)	0.0372(14)	
C_1	0.2417(26)	0.3045(19)	0.3084(44)	0.0725
N_1	0.1248(24)	0.3492(17)	0.3406(40)	0.0452
C_2	0.1627(31)	0.4509(24)	0.5947(51)	0.0948
C_3	0.3373(30)	0.4525(23)	0.7044(50)	0.0890
N_2	0.3674(24)	0.3587(18)	0.5015(40)	0.0474
S_2	−0.1672(7)	0.0845(6)	−0.4088(10)	
S_3	−0.2682(6)	0.2163(5)	−0.2395(10)	
C_4	−0.3930(30)	0.1257(23)	−0.1146(51)	0.0942
O_1	−0.3586(19)	0.2701(14)	−0.4480(31)	0.0623
O_2	−0.1612(21)	0.3160(16)	−0.0235(34)	0.0724

STRUCTURE ANALYSIS

The tellurium atoms lie in centres of symmetry in both dimorphs. In the space group $P\bar{1}$ of I, tellurium contributes to all reflections, whereas in the space group $P2_1/c$ of II, it contributes only to reflections with $k+l$ even. The structures were solved in projections, along the c and a axes of I and along the b and c axes of II, through Fourier summations of all but the weakest reflections, with positive signs. In the c-axis projection of II, the $k+l$ odd reflections were not included in the first Fourier synthesis. The sulphur atoms were located from the first Fourier maps, and the lighter atoms from later maps.

Table 2. Atomic coordinates for monoclinic dimorph, in fractions of cell edges. Standard deviations from least squares are given in parentheses. Isotropic thermal parameters (Å²) in the form $\exp[-8\pi^2 U(\sin^2\theta/\lambda^2)]$.

	x	y	z	U
Te	0	0	0	
S_1	−0.0446(5)	0.1260(5)	0.1943(5)	
C_1	−0.1336(17)	0.2576(18)	0.1041(17)	0.0372
N_1	−0.0960(17)	0.3197(17)	0.0144(17)	0.0481
C_2	−0.1930(18)	0.4284(18)	−0.0481(18)	0.0429
C_3	−0.3044(20)	0.4263(20)	0.0219(20)	0.0489
N_2	−0.2527(14)	0.3143(14)	0.1095(14)	0.0396
S_2	0.3177(4)	0.0274(4)	0.1506(5)	
S_3	0.3506(4)	0.1903(4)	0.0782(5)	
C_4	0.3315(21)	0.1646(22)	−0.0967(21)	0.0556
O_1	0.5110(13)	0.2298(14)	0.1744(13)	0.0516
O_2	0.2279(12)	0.2767(12)	0.0576(12)	0.0473

Table 3. Anisotropic thermal parameters (Å²) in the form $\exp[-2\pi^2(h^2a^{-2}U_{11}+\cdots+2hka^{-1}b^{-1}U_{12}+\cdots)]$. All values have been multiplied by 10^4.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
			Triclinic dimorph			
Te	346	379	416	140	52	87
S_1	543	590	859	115	78	380
S_2	587	886	432	385	124	125
S_3	371	617	506	216	158	31
			Monoclinic dimorph			
Te	394	229	431	−2	9	192
S_1	698	449	522	54	65	362
S_2	445	340	603	2	100	194
S_3	391	348	587	−8	−29	277

In all but the *c*-axis projection of I, some overlapping occurred, and in order to locate the carbon and nitrogen atom of the ethylenethiourea groups, use was also made of models based on known distances and angles.

Three-dimensional least squares refinement was started with coordinates derived from the projections. At first, only the tellurium and sulphur parameters were allowed to vary, and later also the parameters of the lighter atoms. Anisotropic thermal parameters for tellurium and sulphur were introduced at later stages. The last refinement cycles gave shifts which were insignificant relative to the standard deviations. The final value of the conventional *R* factor was 0.091 for I and 0.068 for II.

The final atomic coordinates are listed in Tables 1 and 2, anisotropic thermal parameters in Table 3, and observed and calculated structure factors in Tables 4 and 5.

RESULTS

Bond lengths and angles involving the TeS_4 coordination groups, from the coordinates of Tables 1 and 2, are listed in Table 6. The tellurium atoms being located in crystallographic centres of symmetry, the TeS_4 coordination groups are exactly planar in both dimorphs.

Bond lengths and angles are very like in the two dimorphs. The values of Table 6 differ significantly only in three instances: Te−S(ethylenethiourea) = 2.663(6) Å in I and 2.687(5) Å in II, $\Delta = 3.1\ \sigma(\Delta)$; ∠ S−Te−S = 91.6(2)° in I and 92.6(2)° in II, $\Delta = 3.5\ \sigma(\Delta)$; ∠Te−S−S = 103.9(3)° in I and 102.3(2)° in II, $\Delta = 4.4\ \sigma(\Delta)$.

For the thiourea complex, *trans*-dimethanethiosulphonatobis(thiourea)tellurium(II), the following bond lengths and angles were found:[5] Te−S(thiourea) = 2.667(15) Å, Te−S(thiosulphonate) = 2.684(15) Å, ∠S−Te−S = 90.6(5)°, S−C = 1.76(6) Å, S−S = 2.024(18) Å, ∠ Te−S−C = 100.7(2.1)°,

Table 4. Observed and calculated structure factors (× 10) for triclinic dimorph. Unobserved reflections are indicated by a minus sign on $F(O)$ and included at the threshold values.

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)
0	1	0	433	524	0	2	4	113	-26	1	-2	3	135	138	1	-6	-3	218	203	2-10	2	177	166
0	2	0	73	-23	0	3	4	115	126	1	-1	3	252	252	1	-5	-3	150	121	2	-9	2	327	310
0	3	0	316	264	0	4	4	226	200	1	0	3	241	242	1	-4	-3	-61	19	2	-8	2	321	319
0	4	0	315	357	0	5	4	219	209	1	1	3	71	56	1	-3	-3	346	312	2	-7	2	89	82
0	5	0	337	293	0	6	4	116	126	1	2	3	114	106	1	-2	-3	415	361	2	-6	2	-65	51
0	6	0	264	286	0	7	4	59	75	1	3	3	336	344	1	-1	-3	480	535	2	-5	2	305	303
0	7	0	76	91	0	8	4	62	93	1	4	3	362	336	1	0	-3	527	535	2	-4	2	192	179
0	8	0	63	75	0-10	5	57	68	1	5	3	288	258	1	1	-3	258	243	2	-3	2	132	155
0	9	0	232	220	0	-9	5	73	86	1	6	3	152	157	1	2	-3	243	226	2	-2	2	267	239
0	10	0	168	175	0	-8	5	99	111	1-11	4	51	38	1	3	-3	332	339	2	-1	2	261	284
0	11	0	142	152	0	-7	5	60	65	1-10	4	-51	39	1	4	-3	187	185	2	0	2	478	481
0	12	0	56	75	0	-6	5	-59	36	1	-9	4	80	97	1	5	-3	-57	17	2	1	2	567	515
0-12	1	76	104	0	-5	5	75	663	1	-8	4	162	153	1	6	-3	174	195	2	2	2	358	376
0-11	1	81	72	0	-4	5	125	110	1	-7	4	132	147	1	7	-3	134	128	2	3	2	416	371
0-10	1	148	154	0	-3	5	134	135	1	-6	4	-67	35	1	8	-3	168	157	2	4	2	233	203
0	-9	1	163	172	0	-2	5	148	160	1	-5	4	96	91	1	9	-3	127	121	2	5	2	149	136
0	-8	1	208	225	0	-1	5	148	140	1	-4	4	278	281	1	-8	-4	106	102	2	6	2	123	152
0	-7	1	342	329	0	0	5	209	211	1	-3	4	385	388	1	-7	-4	155	147	2-10	3	-79	22
0	-6	1	440	463	0	1	5	244	265	1	-2	4	321	306	1	-6	-4	201	188	2	-9	3	153	175
0	-5	1	301	267	0	2	5	133	122	1	-1	4	225	217	1	-5	-4	165	165	2	-8	3	303	272
0	-4	1	526	540	0	3	5	80	67	1	0	4	150	157	1	-4	-4	218	193	2	-7	3	257	241
0	-3	1	74	63	0	4	5	112	122	1	1	4	245	244	1	-3	-4	232	207	2	-6	3	118	134
0	-2	1	132	-113	0	5	5	103	103	1	2	4	153	140	1	-2	-4	164	157	2	-5	3	176	186
0	-1	1	335	335	0	6	5	61	65	1	3	4	-67	16	1	-1	-4	113	113	2	-4	3	491	492
0	0	1	290	282	0	-7	6	106	115	1	4	4	116	114	1	0	-4	143	140	2	-3	3	442	424
0	1	1	815	883	0	-6	6	106	132	1	5	4	110	119	1	1	-4	210	204	2	-2	3	152	170
0	2	1	404	-401	0	-5	6	79	72	1	6	4	98	77	1	2	-4	127	93	2	-1	3	-65	9
0	3	1	195	217	0	-4	6	59	49	1-10	5	39	44	1	3	-4	129	130	2	0	3	81	80
0	4	1	230	194	0	-3	6	104	131	1	-9	5	85	84	1	4	-4	123	134	2	1	3	233	238
0	5	1	640	696	0	-2	6	89	91	1	-8	5	151	163	1	5	-4	277	263	2	2	3	368	314
0	6	1	458	448	0	-1	6	-48	32	1	-7	5	167	147	1	6	-4	318	323	2	3	3	93	91
0	7	1	215	186	0	0	6	-46	33	1	-6	5	117	107	1	7	-4	290	283	2	4	3	110	103
0	8	1	74	23	0	1	6	-42	36	1	-5	5	-63	36	1	8	-4	-79	32	2	5	3	214	204
0	9	1	120	121	0	2	6	64	84	1	-4	5	-63	-23	1	9	-4	107	91	2	6	3	168	155
0	10	1	173	167	1-12	0	131	157	1	-3	5	116	129	1	10	-4	143	143	2-10	4	82	89
0	11	1	76	102	1-11	0	110	107	1	-2	5	212	210	1	-6	-5	67	67	2	-9	4	83	62
0	12	1	36	53	1-10	0	80	95	1	-1	5	-65	52	1	-5	-5	98	91	2	-8	4	127	144
0-12	2	134	131	1	-9	0	134	202	1	0	5	93	81	1	-4	-5	102	89	2	-7	4	108	118
0-11	2	151	160	1	-8	0	258	227	1	1	5	143	150	1	-3	-5	78	58	2	-6	4	95	76
0-10	2	75	63	1	-7	0	408	393	1	2	5	194	199	1	-2	-5	81	60	2	-5	4	-77	77
0	-9	2	-67	18	1	-6	0	294	265	1	3	5	170	192	1	-1	-5	151	141	2	-4	4	-75	80
0	-8	2	124	128	1	-5	0	84	93	1	4	5	106	101	1	0	-5	306	312	2	-3	4	262	240
0	-7	2	255	254	1	-4	0	61	31	1	-7	6	72	94	1	1	-5	275	274	2	-2	4	397	366
0	-6	2	342	305	1	-3	0	575	614	1	-6	6	113	139	1	2	-5	119	102	2	-1	4	182	176
0	-5	2	345	363	1	-2	0	151	166	1	-5	6	89	92	1	3	-5	84	87	2	0	4	95	74
0	-4	2	140	111	1	-1	0	353	407	1	-4	6	95	97	1	4	-5	187	202	2	1	4	348	302
0	-3	2	485	557	1	0	0	410	530	1	-3	6	97	109	1	5	-5	184	191	2	2	4	266	263
0	-2	2	491	516	1	1	0	624	632	1	-2	6	103	115	1	6	-5	80	90	2	3	4	171	165
0	-1	2	487	539	1	2	0	906	893	1	-1	6	94	114	1	7	-5	-61	25	2	4	4	-71	55
0	0	2	516	557	1	3	0	398	383	1	0	6	-41	36	1	8	-5	-55	28	2	-9	5	80	95
0	1	2	344	373	1-12	1	123	137	1	1	6	-37	4	1	-9	-6	66	73	2	-8	5	80	84
0	2	2	162	180	1-11	1	139	140	1	2	6	-27	46	1	-8	-6	91	93	2	-7	5	134	127
0	3	2	258	235	1-10	1	70	24	1-12	-1	71	90	1	-7	-6	152	162	2	-6	5	166	180
0	4	2	215	201	1	-9	1	73	44	1-11	-1	77	109	1	0	-6	107	121	2	-5	5	158	156
0	5	2	78	69	1	-8	1	269	258	1-10	-1	107	93	1	1	-6	-55	14	2	-4	5	89	76
0	6	2	257	260	1	-7	1	421	406	1	-9	-1	96	85	1	2	-6	-55	16	2	-3	5	-67	78
0	7	2	210	205	1	-6	1	463	453	1	-8	-1	165	163	1	3	-6	79	55	2	-2	5	-67	98
0	8	2	126	123	1	-5	1	367	372	1	-7	-1	114	120	1	4	-6	77	84	2	-1	5	89	94
0	9	2	135	105	1	-4	1	305	297	1	-6	-1	400	370	1	5	-6	73	97	2	0	5	87	79
0	10	2	122	142	1	-3	1	422	434	1	-5	-1	506	452	1	6	-6	67	72	2	1	5	-67	47
0	11	2	99	128	1	-2	1	874	1099	1	-4	-1	353	402	1	7	-6	71	88	2	2	5	127	121
0-12	3	65	82	1	-1	1	221	245	1	-3	-1	531	486	2-12	0	114	124	2	3	5	157	162
0-11	3	149	137	1	0	1	116	-112	1	-2	-1	391	367	2-11	0	102	113	2	-4	6	103	142
0-10	3	185	174	1	1	1	183	-164	1	-1	-1	560	583	2-10	0	99	99	2	-3	6	129	86
0	-9	3	125	130	1	2	1	233	234	1	0	-1	859	1036	2	-9	0	186	188	2	-2	6	57	90
0	-8	3	-61	31	1	3	1	548	535	1	1	-1	504	509	2	-8	0	434	409	2	-1	6	76	107
0	-7	3	269	249	1	4	1	487	466	1	2	-1	713	-753	2	-7	0	391	379	2	0	6	76	104
0	-6	3	468	463	1	5	1	245	238	1	3	-1	506	546	2	-6	0	302	264	2	1	6	51	73
0	-5	3	208	187	1	6	1	210	220	1	4	-1	567	517	2	-5	0	206	203	2-12	-1	98	124
0	-4	3	83	-49	1-12	2	138	112	1	5	-1	292	267	2	-4	0	66	41	2-11	-1	100	102
0	-3	3	-50	44	1-11	2	147	149	1	6	-1	246	200	2	-3	0	386	374	2-10	-1	93	99
0	-2	3	264	251	1-10	2	183	170	1	7	-1	122	135	2	-2	0	729	842	2	-9	-1	172	171
0	-1	3	332	274	1	-9	2	146	116	1-11	-2	93	96	2	-1	0	355	458	2	-8	-1	312	274
0	0	3	433	424	1	-8	2	82	60	1-10	-2	126	137	2	0	0	397	-480	2	-7	-1	207	201
0	1	3	254	228	1	-7	2	194	182	1	-9	-2	159	146	2	1	0	270	264	2	-6	-1	123	106
0	2	3	248	239	1	-6	2	364	381	1	-8	-2	64	80	2	2	0	689	745	2	-5	-1	148	-107
0	3	3	443	356	1	-5	2	84	108	1	-7	-2	322	276	2	3	0	711	768	2	-4	-1	367	372
0	4	3	417	400	1	-4	2	103	-80	1	-6	-2	530	492	2	4	0	381	372	2	-3	-1	886	916
0	5	3	218	207	1	-3	2	389	433	1	-5	-2	363	329	2-12	1	71	72	2	-2	-1	912	985
0	6	3	110	105	1	-2	2	678	777	1	-4	-2	-82	37	2-11	1	87	100	2	-1	-1	201	187
0	7	3	106	90	1	-1	2	584	630	1	-3	-2	246	-175	2-10	1	145	145	2	0	-1	413	478
0	8	3	73	82	1	0	2	417	432	1	-2	-2	434	413	2	-9	1	184	162	2	1	-1	486	521
0	9	3	79	99	1	1	2	359	327	1	-1	-2	455	397	2	-8	1	124	126	2	2	-1	502	530
0	10	3	46	72	1	2	2	77	78	1	0	-2	333	394	2	-7	1	230	208	2	3	-1	311	329
0-11	4	62	59	1	3	2	524	414	1	1	-2	144	157	2	-6	1	348	324	2	4	-1	239	131
0-10	4	83	64	1	4	2	467	478	1	2	-2	407	384	2	-5	1	326	306	2	5	-1	233	238
0	-9	4	81	96	1	5	2	77	112	1	3	-2	353	334	2	-4	1	308	270	2	6	-1	165	147
0	-8	4	87	73	1-12	3	50	64	1	4	-2	669	730	2	-3	1	613	659	2	7	-1	221	197
0	-7	4	90	96	1-11	3	67	59	1	5	-2	479	473	2	-2	1	780	912	2-10	-2	81	73
0	-6	4	248	243	1-10	3	109	101	1	6	-2	71	56	2	-1	1	395	542	2	-9	-2	184	166
0	-5	4	365	332	1	-9	3	203	217	1	7	-2	155	174	2	0	1	213	267	2	-8	-2	265	252
0	-4	4	327	307	1	-8	3	252	240	1	8	-2	165	159	2	1	1	268	265	2	-7	-2	281	245
0	-3	4	139	157	1	-7	3	191	167	1	9	-2	206	182	2	2	1	137	119	2	-6	-2	173	157
0	-2	4	130	116	1	-6	3	242	254	1-10	-3	39	75	2	3	1	225	206	2	-5	-2	486	443
0	-1	4	333	313	1	-5	3	344	322	1	-9	-3	53	39	2	4	1	447	425	2	-4	-2	426	371
0	0	4	292	279	1	-4	3	211	212	1	-8	-3	77	81	2	5	1	105	95	2	-3	-2	428	419
0	1	4	-59	11		-3	3	159	153	1	-7	-3	203	181	2-11	2	83	77	2	-2	-2	440	364

Table 4. 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	-1	-2	105	101	-7	2	0	194	189	-5	8	0	244	242	7	3	1	100	81	-2	12	1	70	124
2	0	-2	163	162	-6	2	0	611	560	-4	8	0	83	75	8	3	1	-74	21	-1	12	1	65	93
2	1	-2	136	135	-5	2	0	572	565	-3	8	0	185	179	9	3	1	103	120	-3	12	1	592	634
2	2	-2	-64	22	-4	2	0	115	131	-2	8	0	416	409	-10	4	1	-75	34	3	-1	1	261	300
2	3	-2	308	295	-3	2	0	63	102	1	8	0	-46	73	-9	4	1	142	124	4	-1	1	311	349
2	4	-2	700	757	3	2	0	396	376	2	8	0	269	239	-8	4	1	241	228	5	-1	1	424	428
2	5	-2	237	227	4	2	0	72	59	3	8	0	276	287	-7	4	1	305	306	6	-1	1	219	203
2	6	-2	248	237	5	2	0	142	162	4	8	0	-55	72	-6	4	1	359	333	7	-1	1	-81	28
2	7	-2	220	214	6	2	0	362	369	5	8	0	-48	47	-5	4	1	350	343	8	-1	1	175	143
2	8	-2	190	160	7	2	0	229	216	6	8	0	111	136	-4	4	1	311	297	9	-1	1	297	280
2	-10	-3	92	105	8	2	0	106	115	7	8	0	70	84	-3	4	1	125	123	10	-1	1	171	175
2	-9	-3	127	140	9	2	0	147	139	-9	9	0	36	59	3	4	1	148	130	-4	-2	1	272	291
2	-8	-3	127	114	10	2	0	95	93	-8	9	0	222	227	4	4	1	93	100	-3	-2	1	922	1045
2	-7	-3	144	128	-11	3	0	120	126	-7	9	0	310	286	5	4	1	244	262	3	-2	1	432	377
2	-6	-3	264	252	-10	3	0	124	143	-6	9	0	100	102	6	4	1	263	253	4	-2	1	174	192
2	-5	-3	130	107	-9	3	0	256	224	-5	9	0	-55	15	7	4	1	-77	83	5	-2	1	249	277
2	-4	-3	-71	-4	-8	3	0	156	144	-4	9	0	163	164	8	4	1	-66	33	6	-2	1	225	214
2	-3	-3	207	175	-7	3	0	149	132	-3	9	0	238	232	-9	5	1	197	169	7	-2	1	-81	29
2	-2	-3	363	353	-6	3	0	373	355	1	9	0	149	150	-8	5	1	299	307	8	-2	1	227	221
2	-1	-3	530	504	-5	3	0	312	337	2	9	0	86	85	-7	5	1	192	176	9	-2	1	332	308
2	0	-3	215	195	-4	3	0	292	338	3	9	0	88	76	-6	5	1	186	183	10	-2	1	111	102
2	1	-3	284	320	-3	3	0	283	290	4	9	0	91	108	-5	5	1	251	283	-4	-3	1	368	369
2	2	-3	330	316	3	3	0	271	265	5	9	0	115	118	-4	5	1	504	480	-3	-3	1	497	463
2	3	-3	436	458	4	3	0	163	-113	6	9	0	68	91	-3	5	1	244	232	3	-3	1	204	218
2	4	-3	403	424	5	3	0	265	239	-8	10	0	123	121	3	5	1	186	178	4	-3	1	300	294
2	5	-3	266	276	6	3	0	398	372	-7	10	0	251	229	4	5	1	213	204	5	-3	1	294	297
2	6	-3	133	-64	7	3	0	206	211	-6	10	0	91	120	5	5	1	144	163	6	-3	1	169	154
2	7	-3	-75	27	8	3	0	111	100	-5	10	0	50	65	6	5	1	151	133	7	-3	1	403	358
2	8	-3	245	233	9	3	0	93	93	-4	10	0	199	203	7	5	1	126	144	8	-3	1	293	291
2	9	-3	150	143	10	3	0	111	134	-3	10	0	194	191	8	5	1	83	104	9	-3	1	-77	83
2	-8	-4	79	102	-11	4	0	219	190	1	10	0	194	187	-10	6	1	117	147	10	-3	1	78	90
2	-7	-4	164	183	-10	4	0	118	100	2	10	0	-48	52	-9	6	1	-77	74	11	-3	1	107	109
2	-6	-4	216	176	-9	4	0	174	156	3	10	0	-48	40	-8	6	1	175	147	-5	-4	1	363	365
2	-5	-4	155	148	-8	4	0	265	252	4	10	0	124	139	-7	6	1	293	294	-4	-4	1	396	379
2	-4	-4	156	144	-7	4	0	84	106	5	10	0	81	112	-6	6	1	196	197	-3	-4	1	84	42
2	-3	-4	293	253	-6	4	0	-51	58	-7	11	0	91	104	-5	6	1	372	369	3	-4	1	641	632
2	-2	-4	445	426	-5	4	0	396	394	-6	11	0	181	179	-3	6	1	121	99	4	-4	1	327	299
2	-1	-4	228	217	-4	4	0	731	746	-5	11	0	138	148	2	6	1	-77	-7	5	-4	1	111	-126
2	0	-4	124	131	-3	4	0	491	499	-4	11	0	48	66	3	6	1	198	193	6	-4	1	122	104
2	1	-4	-85	-11	1	4	0	538	543	-3	11	0	-32	102	4	6	1	293	291	7	-4	1	471	456
2	2	-4	124	94	3	4	0	384	396	1	11	0	100	112	5	6	1	115	101	8	-4	1	292	270
2	3	-4	304	321	4	4	0	226	221	2	11	0	48	70	6	6	1	-75	78	9	-4	1	75	41
2	4	-4	183	203	5	4	0	73	58	3	11	0	32	70	7	6	1	110	135	10	-4	1	77	80
2	5	-4	-75	26	6	4	0	199	209	-6	12	0	122	126	8	6	1	76	107	11	-4	1	150	130
2	6	-4	110	114	7	4	0	235	209	-5	12	0	131	145	-10	7	1	220	241	-5	-5	1	457	459
2	7	-4	214	210	8	4	0	115	108	-4	12	0	41	70	-9	7	1	103	95	-4	-5	1	414	387
2	8	-4	169	153	9	4	0	77	95	-3	12	0	35	37	-8	7	1	-98	-21	-3	-5	1	-86	13
2	9	-4	135	144	10	4	0	72	93	1	12	0	-28	50	-7	7	1	117	143	3	-5	1	505	497
2	-7	-5	99	133	-11	5	0	131	143	-8	0	1	316	346	-6	7	1	413	402	4	-5	1	294	274
2	-6	-5	99	104	-10	5	0	174	150	-7	0	1	93	89	-5	7	1	342	342	5	-5	1	74	76
2	-5	-5	77	51	-9	5	0	115	128	-6	0	1	83	-30	-4	7	1	157	132	6	-5	1	317	314
2	-4	-5	-70	48	-8	5	0	319	280	-5	0	1	420	461	-3	7	1	210	191	7	-5	1	255	205
2	-3	-5	128	121	-7	5	0	342	280	-4	0	1	556	639	1	7	1	304	304	8	-5	1	153	149
2	-2	-5	106	112	-6	5	0	-93	-3	-3	0	1	172	155	2	7	1	272	277	9	-5	1	166	155
2	-1	-5	-75	00	-5	5	0	118	114	3	0	1	515	564	3	7	1	144	152	10	-5	1	95	110
2	0	-5	95	72	-4	5	0	550	591	4	0	1	571	615	4	7	1	-81	87	-5	-6	1	201	207
2	1	-5	142	143	-3	5	0	527	571	5	0	1	159	155	5	7	1	-76	75	-4	-6	1	175	162
2	2	-5	253	249	1	5	0	443	419	6	0	1	132	119	6	7	1	93	85	-3	-6	1	227	204
2	3	-5	192	178	2	5	0	170	165	7	0	1	290	269	-10	8	1	167	209	3	-6	1	84	23
2	4	-5	156	150	3	5	0	296	276	8	0	1	100	112	-9	8	1	139	173	4	-6	1	89	-5
2	5	-5	133	140	4	5	0	425	424	9	0	1	136	120	-8	8	1	-75	24	5	-6	1	453	404
2	6	-5	139	123	5	5	0	170	162	10	0	1	145	161	-7	8	1	113	113	6	-6	1	408	372
2	7	-5	86	94	6	5	0	-55	25	-9	1	1	151	161	-6	8	1	208	205	7	-6	1	155	153
2	-4	-6	74	88	7	5	0	-55	79	-8	1	1	280	279	-5	8	1	144	162	8	-6	1	160	155
2	-3	-6	95	106	8	5	0	145	161	-7	1	1	274	266	-4	8	1	116	142	9	-6	1	125	104
2	-2	-6	97	109	9	5	0	106	127	-6	1	1	286	251	-3	8	1	252	256	10	-6	1	68	75
2	-1	-6	122	136	-11	6	0	57	63	-5	1	1	332	308	1	8	1	185	181	-4	-7	1	116	104
2	0	-6	130	170	-10	6	0	188	199	-4	1	1	304	343	2	8	1	303	307	-3	-7	1	298	284
2	1	-6	102	138	-9	6	0	321	253	-3	1	1	406	469	3	8	1	-81	83	3	-7	1	73	-21
2	2	-6	-65	32	-8	6	0	179	172	3	1	1	662	731	4	8	1	-75	25	4	-7	1	234	211
2	3	-6	-84	36	-7	6	0	167	145	4	1	1	386	401	5	8	1	113	110	5	-7	1	476	422
2	4	-6	78	83	-6	6	0	215	206	5	1	1	138	193	6	8	1	89	109	6	-7	1	176	166
2	5	-6	74	87	-5	6	0	124	142	6	1	1	96	69	-9	9	1	117	136	7	-7	1	100	105
3	0	0	226	233	-4	6	0	276	268	7	1	1	231	193	-8	9	1	150	109	8	-7	1	233	200
4	0	0	260	314	-3	6	0	436	431	8	1	1	222	211	-7	9	1	-75	75	9	-7	1	104	124
5	0	0	422	422	1	6	0	230	277	9	1	1	87	62	-6	9	1	-79	74	-5	-8	1	203	190
6	0	0	239	247	2	6	0	210	215	10	1	1	76	91	-5	9	1	223	218	-2	-8	1	173	170
7	0	0	276	269	3	6	0	129	115	-9	2	1	117	122	-4	9	1	226	221	-1	-8	1	179	155
8	0	0	330	315	4	6	0	301	283	-8	2	1	231	218	-3	9	1	203	211	5	-8	1	179	163
9	0	0	63	67	5	6	0	201	211	-7	2	1	417	406	1	9	1	139	137	6	-8	1	301	269
10	0	0	-48	25	6	6	0	-55	23	-6	2	1	321	332	2	9	1	134	148	5	-9	1	203	201
11	0	0	129	139	7	6	0	102	94	-5	2	1	-58	44	3	9	1	131	133	6	-8	1	193	190
-11	1	0	113	133	8	6	0	134	157	-4	2	1	141	-56	4	9	1	77	65	7	-8	1	193	197
-10	1	0	234	224	-10	7	0	154	156	-3	2	1	628	604	5	9	1	70	76	-3	-9	1	99	79
-9	1	0	136	116	-9	7	0	310	276	3	2	1	337	365	-8	10	1	93	142	-2	-9	1	203	173
-8	1	0	-55	19	-8	7	0	113	114	4	2	1	401	408	-7	10	1	111	119	-1	-9	1	327	277
-7	1	0	233	196	-7	7	0	-55	58	5	2	1	351	329	-6	10	1	71	33	2	-9	1	203	162
-6	1	0	305	324	-6	7	0	237	275	6	2	1	99	123	-5	10	1	129	139	3	-9	1	275	244
-5	1	0	532	540	-5	7	0	296	279	7	2	1	117	115	-4	10	1	211	233	4	-9	1	144	152
-4	1	0	561	484	-4	7	0	129	126	8	2	1	184	172	-3	10	1	136	160	5	-9	1	116	105
-3	1	0	188	169	-3	7	0	197	223	9	2	1	94	96	1	10	1	101	123	6	-9	1	210	218
3	1	0	317	357	1	7	0	-55	6	10	2	1	79	75	2	10	1	81	76	7	-9	1	224	226
4	1	0	511	477	2	7	0	310	305	-10	3	1	187	177	3	10	1	81	104	-2	-10	1	134	128
5	1	0	258	241	3	7	0	200	274	-9	3	1	101	90	4	10	1	61	84	-1	-10	1	283	239
6	1	0	111	124	4	7	0	113	131	-8	3	1	-82	70	-7	11	1	84	113	2	-10	1	163	143
7	1	0	321	304	5	7	0	113	122	-7	3	1	451	451	-6	11	1	-56	43	3	-10	1	236	214
8	1	0	332	298	6	7	0	104	118	-6	3	1	431	515	-5	11	1	77	69	4	-10	1	159	128
9	1	0	-164	85	7	7	0	102	96	-5	3	1	220	201	-4	11	1	132	175	5	-10	1	171	153
10	1	0	-46	1	8	7	0	48	63	-4	3	1	96	-61	-3	11	1	117	147	6	-10	1	159	163
11	1	0	88	87	-10	8	0	70	70	-3	3	1	199	167	-1	11	1	149	109	-2	-11	1	81	100
-11	2	0	93	92	-9	8	0	149	151	3	3	1	78	72	1	11	1	69	96	-1	-11	1	30	35
-10	2	0	298	263	-8	8	0	285	241	4	3	1	200	177	-5	12	1	61	93	2	-11	1	91	100
-9	2	0	172	163	-7	8	0	142	155	5	3	1	397	359	-4	12	1	59	65	3	-11	1	90	87
-8	2	0	120	-66	-6	8	0	133	146	6	3	1	412	355	-3	12	1	70	100	4	-11	1	229	206
																				5	-11	1	175	184

Table 5. Observed and calculated structure factors (× 10) for monoclinic dimorph. Unobserved reflections are indicated by a minus sign on $F(O)$ and included at the threshold values.

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	885	968	7	0-10	326	323	0	1 11	207	239	4	1 -9	723	666	0	2 8	527	511				
2	0	0	273	298	8	0-10	542	523	1	1 11	201	206	5	1 -9	665	641	1	2 8	-80	91				
3	0	0	1513	1626	9	0-10	-69	61	1	1 -1	1147	1072	6	1 -9	285	298	2	2 8	380	396				
4	0	0	724	701	10	0-10	-61	18	2	1 -1	571	470	7	1 -9	537	511	3	2 8	312	324				
5	0	0	193	177	11	0-10	236	277	3	1 -1	1025	1052	8	1 -9	440	437	0	2 9	177	204				
6	0	0	861	849	1	0-12	87	-107	4	1 -1	706	721	9	1 -9	125	162	1	2 9	-75	86				
7	0	0	127	94	2	0-12	155	172	5	1 -1	170	157	10	1 -9	330	335	2	2 9	-68	59				
8	0	0	250	210	3	0-12	230	243	6	1 -1	439	489	11	1 -9	305	293	3	2 9	-56	90				
9	0	0	306	405	4	0-12	109	-89	7	1 -1	429	411	1	1-10	134	173	0	2 10	-71	95				
10	0	0	133	111	5	0-12	91	99	8	1 -1	181	176	2	1-10	103	88	1	2 10	201	161				
0	0	2	1433	1416	6	0-12	248	293	9	1 -1	247	262	3	1-10	-66	-60	2	2 10	258	266				
1	0	2	330	338	7	0-12	-62	-4	10	1 -1	234	238	4	1-10	132	123	0	2 11	-56	43				
2	0	2	822	774	8	0-12	252	272	11	1 -1	105	117	5	1-10	148	153	1	2 11	85	-90				
3	0	2	927	862	9	0-12	256	236	1	1 -2	276	-244	6	1-10	104	-111	1	2 -1	641	-556				
4	0	2	135	155	1	1 0	169	-158	2	1 -2	360	275	7	1-10	165	201	2	2 -1	890	880				
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Table 5. Continued.

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2	2	-9	164	-158
3	2	-9	186	-178
4	2	-9	5	0
5	2	-9	-80	-97
6	2	-9	-80	23
7	2	-9	277	250
8	2	-9	-79	24
9	2	-9	-75	37
10	2	-9	141	168
11	2	-9	-54	18
1	2	-10	386	365
2	2	-10	322	330
3	2	-10	210	221
4	2	-10	478	450
5	2	-10	355	348
6	2	-10	286	270
7	2	-10	253	223
8	2	-10	184	231
9	2	-10	225	270
10	2	-10	235	222
11	2	-10	140	144
1	2	-11	112	96
2	2	-11	221	215
3	2	-11	-73	14
4	2	-11	106	120
5	2	-11	237	223
6	2	-11	-74	-6
7	2	-11	102	110
8	2	-11	-68	72
9	2	-11	131	-136
10	2	-11	-52	-15
1	2	-12	174	212
2	2	-12	231	232
3	2	-12	238	221
4	2	-12	201	203
5	2	-12	210	233
6	2	-12	170	169
7	2	-12	201	153
8	2	-12	100	124
9	2	-12	269	283
10	2	-12	173	191
3	2	-13	63	-102
4	2	-13	122	-123
5	2	-13	-48	-2
6	2	-13	96	-106
7	2	-13	103	-124
0	2	C	188	162
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2	2	C	648	626
3	2	C	1141	1142
4	2	C	657	671
5	2	C	539	476
6	2	C	573	572
7	2	C	648	642
8	2	C	-58	4
9	2	C	235	242
10	2	C	350	362
1	3	C	349	297
2	3	C	-33	64
3	3	C	-38	-21
4	3	C	-43	65
5	3	C	185	148
6	3	C	337	379
7	3	C	-58	75
8	3	C	-57	-4
9	3	C	-50	49
10	3	C	-37	-26
0	4	C	964	1032
1	4	C	240	150
2	4	C	279	293
3	4	C	469	437
4	4	C	543	552
5	4	C	255	284
6	4	C	641	610
7	4	C	402	407
8	4	C	327	334
9	4	C	347	367
10	4	C	252	266
1	5	C	125	-129
2	5	C	101	-56
3	5	C	-45	23
4	5	C	293	-256
5	5	C	266	-261
6	5	C	82	-43
7	5	C	92	-84
8	5	C	-34	-53
9	5	C	-44	59
C	6	C	780	883
1	6	C	542	531
2	6	C	486	470
3	6	C	1037	1042
4	6	C	322	323
5	6	C	336	337
6	6	C	437	464
7	6	C	217	223
8	6	C	226	242
9	6	C	338	365
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2	7	O	365	362
3	7	O	177	-173
4	7	O	403	-358
5	7	O	254	263
6	7	O	-58	-43
7	7	O	281	-265
8	7	O	73	73
9	7	O	-32	-32
0	8	O	418	409
1	8	O	754	772
2	8	O	726	811
3	8	O	358	350
4	8	O	603	592
5	8	O	344	361
6	8	O	244	251
7	8	O	372	376
8	8	O	190	166
1	9	O	115	-95
2	9	O	-58	44
3	9	O	242	251
4	9	O	-58	-16
5	9	O	-56	-25
6	9	O	198	201
7	9	O	-43	-17
8	9	O	-29	-19
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2	10	O	306	327
3	10	O	429	453
4	10	O	246	261
5	10	O	291	304
6	10	O	264	259
7	10	O	134	124
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3	11	O	-53	76
4	11	O	-49	-73
5	11	O	185	174
6	11	O	54	70
0	12	O	195	181
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2	12	O	249	240
3	12	O	134	143
4	12	O	221	212
5	12	O	222	225
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1	2	1	1120	-1074
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3	2	1	290	-275
4	2	1	241	-231
5	2	1	311	294
6	2	1	-79	-75
7	2	1	-83	63
8	2	1	219	226
9	2	1	-46	27
10	2	1	-49	43
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2	3	1	599	543
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4	3	1	511	476
5	3	1	284	254
6	3	1	278	247
7	3	1	293	281
8	3	1	174	174
9	3	1	314	318
10	3	1	-36	210
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2	4	1	206	157
3	4	1	244	240
4	4	1	-69	95
5	4	1	132	-83
6	4	1	164	136
7	4	1	165	-181
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9	4	1	146	124
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1	5	1	236	-170
2	5	1	597	609
3	5	1	856	827
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6	5	1	570	611
7	5	1	-31	98
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9	5	1	260	251
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2	6	1	425	423
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4	6	1	134	-152
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8	6	1	-65	86
9	6	1	120	-105
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6	7	1	355	397
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6	8	1	-76	-25
7	8	1	-64	-89
8	8	1	112	105
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6	9	1	296	301
7	9	1	285	263
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5	11	1	238	227
6	11	1	164	182
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3	12	1	104	-121
4	12	1	-51	-17
5	12	1	-32	103
0	13	1	80	102
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3	13	1	68	83
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3	3	-1	629	547
4	3	-1	1299	1233
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7	4	-1	-82	-10
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7	5	-1	405	422
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6	7	-1	203	208
7	7	-1	338	337
8	7	-1	141	140
9	7	-1	150	143
1	8	-1	178	-136
2	8	-1	109	-83
3	8	-1	238	247
4	8	-1	116	-116
5	8	-1	117	-80
6	8	-1	-81	94
7	8	-1	-75	15
8	8	-1	-63	-34
9	8	-1	103	94
1	9	-1	478	445
2	9	-1	501	483
3	9	-1	339	328
4	9	-1	347	353
5	9	-1	181	189
6	9	-1	186	206
7	9	-1	263	239
8	9	-1	197	178
1	10	-1	242	181
2	10	-1	117	99
3	10	-1	183	190
4	10	-1	113	108
5	10	-1	-75	-64
6	10	-1	177	166
7	10	-1	-54	-12
8	10	-1	-26	-137
1	11	-1	333	324
2	11	-1	283	278
3	11	-1	451	415
4	11	-1	284	247
5	11	-1	394	380
6	11	-1	408	386
7	11	-1	-33	168
1	12	-1	229	-201
2	12	-1	-69	44
3	12	-1	113	134
4	12	-1	228	-197
5	12	-1	-49	6
6	12	-1	-26	48
1	13	-1	228	196
2	13	-1	435	428
3	13	-1	204	186
4	13	-1	227	256
0	0	2	1308	1419
1	0	2	420	338
2	0	2	780	774
3	0	2	868	862
4	0	2	236	195
5	0	2	930	937
6	0	2	415	377
7	0	2	212	108
8	0	2	558	523
9	0	2	426	381
0	3	2	324	297
1	3	2	196	170
2	3	2	392	-370
3	3	2	627	-607
4	3	2	-104	-110
5	3	2	-115	-69
6	3	2	-122	-28
7	3	2	-120	-39
8	3	2	-106	57
9	3	2	-78	-7
0	4	2	862	912
1	4	2	681	707
2	4	2	692	957
3	4	2	649	701
4	4	2	266	217
5	4	2	511	505
6	4	2	245	209
7	4	2	-118	95
8	4	2	293	254
9	4	2	224	218
0	5	2	243	-242
1	5	2	421	457
2	5	2	-95	102
3	5	2	-103	47
4	5	2	255	221
5	5	2	210	167
6	5	2	-122	-74
7	5	2	-113	73
8	5	2	-93	80
0	6	2	634	643
1	6	2	402	406
2	6	2	791	847
3	6	2	433	406
4	6	2	206	195
5	6	2	514	503
6	6	2	396	386
7	6	2	-106	112
8	6	2	349	306
0	7	2	257	-236
1	7	2	323	318
2	7	2	463	472
3	7	2	278	-270
4	7	2	193	181
5	7	2	243	246
6	7	2	161	-139
7	7	2	-96	72
0	8	2	-114	72
1	8	2	449	490
2	8	2	379	370
3	8	2	419	431
4	8	2	442	459
5	8	2	274	244
6	8	2	359	333
7	8	2	377	374
0	9	2	-121	-109
1	9	2	-122	-65
2	9	2	245	-219
3	9	2	-122	-43
4	9	2	-117	-46
5	9	2	-107	-58
6	9	2	-90	-67
0	10	2	468	485
1	10	2	244	246
2	10	2	414	409
3	10	2	355	335
4	10	2	237	216
5	10	2	325	340
6	10	2	239	223
0	11	2	273	-261
1	11	2	-114	-10
2	11	2	-109	54
3	11	2	238	-230
4	11	2	-89	0
5	11	2	-69	43
0	12	2	390	399
1	12	2	404	426
2	12	2	251	221
3	12	2	291	248
4	12	2	251	257
1	3	-2	618	644
2	3	-2	202	-189
3	3	-2	161	113
4	3	-2	193	-103
5	3	-2	282	-241
6	3	-2	-107	-87
7	3	-2	203	158
8	3	-2	407	-346
9	3	-2	-118	-84
10	3	-2	-100	51
11	3	-2	-67	-56
1	4	-2	1483	1614
2	4	-2	627	645
3	4	-2	732	737
4	4	-2	1120	1169
5	4	-2	822	868
6	4	-2	526	492
7	4	-2	600	595
8	4	-2	387	333
9	4	-2	198	170
10	4	-2	258	237
11	4	-2	151	140
1	5	-2	-82	-80
2	5	-2	338	-300
3	5	-2	-90	63
4	5	-2	276	211
5	5	-2	353	-326
6	5	-2	284	257
7	5	-2	344	290
8	5	-2	-121	12
9	5	-2	-109	90
10	5	-2	172	129
1	6	-2	698	671
2	6	-2	648	575
3	6	-2	782	759
4	6	-2	582	594
5	6	-2	-114	166
6	6	-2	351	339
7	6	-2	388	372
8	6	-2	351	310
9	6	-2	304	292
10	6	-2	221	194
1	7	-2	254	-202
2	7	-2	182	-163
3	7	-2	467	420
4	7	-2	-114	-19
5	7	-2	169	88
6	7	-2	174	145
7	7	-2	-120	-17
8	7	-2	-110	9
9	7	-2	-10	58
1	8	-2	340	296
2	8	-2	460	431
3	8	-2	-117	33
4	8	-2	390	356
5	8	-2	519	533
6	8	-2	-121	113
7	8	-2	370	318
8	8	-2	337	347
9	8	-2	118	114
1	9	-2	191	153
2	9	-2	192	-156
3	9	-2	-122	-37
4	9	-2	-123	99
5	9	-2	331	-296
6	9	-2	-115	25
7	9	-2	-103	116
8	9	-2	230	-219
1	10	-2	491	427
2	10	-2	335	278
3	10	-2	530	526
4	10	-2	376	352
5	10	-2	160	168
6	10	-2	394	362
7	10	-2	222	214
8	10	-2	203	162
1	11	-2	-117	-120
2	11	-2	-116	-124
3	11	-2	253	160
4	11	-2	-108	-46
5	11	-2	-99	-75
6	11	-2	-85	43
1	12	-2	326	281
2	12	-2	430	409
3	12	-2	398	376
4	12	-2	268	252
5	12	-2	333	309
6	12	-2	240	285

Table 6. Dimensions of the coordination group in *trans*-$Te(etu)_2(S_2O_2CH_3)_2$. Standard deviations in parentheses.

	Triclinic dimorph	Monoclinic dimorph
∠ S–Te–S	91.6(2)°	92.6(2)°
Ethylenethiourea ligand		
Te–S	2.663(6) Å	2.687(5) Å
S–C	1.736(22)	1.701(18)
∠ Te–S–C	101.0(8)°	102.5(7)°
Methanethiosulphonate ligand		
Te–S	2.694(6) Å	2.685(4) Å
S–S	2.014(9)	2.015(7)
∠ Te–S–S	103.9(3)°	102.3(2)°

∠Te–S–S = 101.2(7)°. The only significant differences between these values and those of Table 6 occur in the S–Te–S angle relative to the angle in the monoclinic dimorph, and in the Te–S–S angle relative to the angle in the triclinic dimorph. Even these differences are not large.

As in the thiourea complex,[5] the methanethiosulphonate S–S bond length, 2.014(9) Å and 2.015(7) Å, is closer to the length in ionic sodium methanethio-

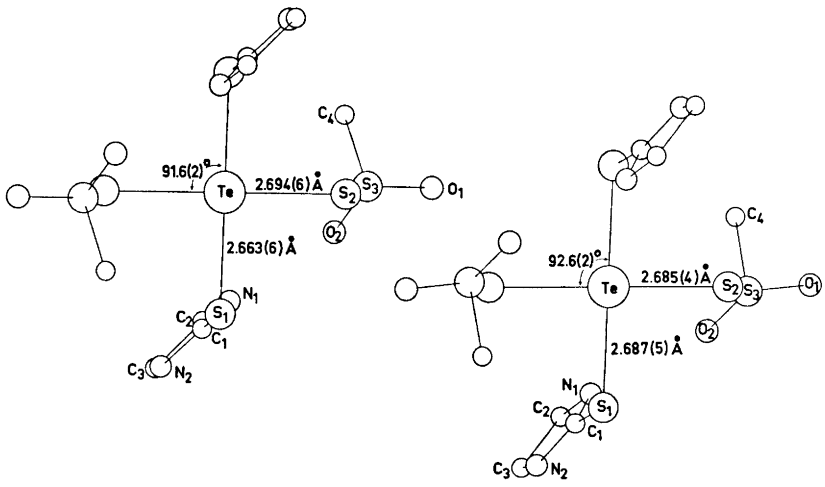

Fig. 1. *trans*-Dimethanethiosulphonatobis(ethylenethiourea)tellurium(II), as seen normal to the TeS_4 coordination group. Left: triclinic dimorph; right: monoclinic dimorph. Tellurium atoms in centres of symmetry.

Table 7. Bond lengths and angles in the ligand groups. Standard deviations in parentheses.

	Triclinic dimorph	Monoclinic dimorph
Ethylenethiourea ligand		
C_1-N_1	1.308(35) Å	1.356(28) Å
C_1-N_2	1.331(26)	1.326(24)
N_1-C_2	1.504(30)	1.449(25)
N_2-C_3	1.456(35)	1.464(25)
C_2-C_3	1.604(39)	1.577(33)
$\angle S_1-C_1-N_1$	124.0(1.3)°	125.6(1.5)°
$\angle S_1-C_1-N_2$	121.5(1.7)°	124.9(1.6)°
$\angle N_1-C_1-N_2$	114.5(1.8)°	109.5(1.6)°
$\angle C_1-N_1-C_2$	111.8(1.8)°	113.3(1.8)°
$\angle C_1-N_2-C_3$	109.7(2.0)°	112.8(1.7)°
$\angle N_1-C_2-C_3$	99.4(1.9)°	101.7(1.6)°
$\angle N_2-C_3-C_2$	104.7(1.8)°	102.6(1.5)°
Methanethiosulphonate ligand		
S_3-O_1	1.457(17) Å	1.439(11) Å
S_3-O_2	1.451(15)	1.437(13)
S_3-C_4	1.732(30)	1.807(24)
$\angle S_2-S_3-O_1$	107.5(0.8)°	107.6(0.6)°
$\angle S_2-S_3-O_2$	111.2(0.9)°	111.1(0.6)°
$\angle S_2-S_3-C_4$	107.9(0.9)°	107.8(0.6)°
$\angle O_1-S_3-O_2$	115.8(0.9)°	115.4(0.8)°
$\angle O_1-S_3-C_4$	105.7(1.2)°	108.4(0.9)°
$\angle O_2-S_3-C_4$	108.3(1.1)°	106.3(0.8)°

sulphonate monohydrate [10] than in the parent substance, tellurium dimethanethiosulphonate.[2]

The values derived for bond lengths and angles in the ethylenethiourea and methanethiosulphonate ligands, other than those associated with the coordinating atoms, are listed in Table 7. In neither dimorph, within the rather large uncertainties, do they differ significantly from values found for these groups in other compounds.

The ethylenethiourea groups are planar within the errors. The largest deviation of the atoms from the least-squares planes of the groups, with the sulphur coordinates, in the calculations of the planes, given three times the weight of the carbon and nitrogen coordinates, are 0.024 Å for C_1 in I and 0.010 Å for C_3 and N_2 in II.

There are some small differences in the rotational positions of the ligand groups, *cf.* Fig. 1 which shows, for the two dimorphs, the molecule projected on to the plane of the TeS_4 coordination group. The plane through the tellurium atom and the two sulphur atoms of the methanethiosulphonate groups makes an angle of 85.2° with the TeS_4 plane in I and 92.8° in II, and the least-squares

plane of the ethylenethiourea group makes an angle of 91.9° with the TeS_4 plane in I and 98.6° in II.

The N–H atoms of the ethylenethiourea groups form hydrogen bonds to oxygen atoms of the methanethiosulphonate groups. One such contact occurs within the molecule: $N_1-H\cdots O_2 = 2.83$ Å in I and 2.95 Å in II, $\angle C_1-N_1\cdots O_2 = 130°$ in I and 116° in II. Another occurs between molecules: $N_2-H\cdots O_1' = 2.85$ Å in I and 2.83 Å in II, $\angle C_1-N_2\cdots O_1' = 125°$ in I and 132° in II, where O_1' is located at $(1+x,y, 1+z)$ relative to O_1 in I and at $(x-1,y,z)$ relative to O_1 in II. All four nitrogen atoms, and all four oxygen atoms of the centrosymmetric molecules, thus engage in hydrogen bonding.

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