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The Crystal and Molecular Structure of a *trans* Square-Planar Complex of Tellurium Dibenzenethiosulphonate with Tetramethylthiourea

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The crystal and molecular structure of *trans*-dibenzenethiosulphonatobis(tetramethylthiourea)tellurium(II), $Te(SC[N(CH_3)_2]_2)_2(S_2O_2C_6H_5)_2$, has been determined by three-dimensional X-ray diffraction methods. The space group is $P2_1/c$ (No. 14) with $Z=2$, and with cell dimensions (standard deviations in parentheses), $a=10.254(5)$ Å, $b=10.029(8)$ Å, $c=15.929(7)$ Å, and $\beta=110.35(5)°$. Based on the intensities of 1251 independent, non-zero reflections, collected by integrating Weissenberg techniques, least squares refinement procedures resulted in a conventional R value of 0.075.

The tellurium atom is bonded to two tetramethylthiourea sulphur atoms and to two benzenethiosulphonate sulphur atoms in a *trans* square-planar arrangement. Bond lengths are: Te – S(tetramethylthiourea) = 2.724(6) Å, Te – S (benzenethiosulphonate) = 2.657(4) Å, and benzenethiosulphonate S – S = 2.025(6) Å. The value of the S – Te – S angle, 79.51(15)°, implies a marked deviation from a strictly square TeS_4 coordination group.

A remarkable feature of *trans*-dibenzenethiosulphonatobis(tetramethylthiourea)tellurium(II), $Te(SC[N(CH_3)_2]_2)_2(S_2O_2C_6H_5)_2$, as compared with other complexes of divalent tellurium thiosulphonates .with thioureas, is its red colour.[1] All other complexes of this type have been found to be yellow. The structures of three yellow complexes have been reported;[2-4] they all display a *trans* square-planar coordination, with four sulphur atoms bonded to the central tellurium atom. The benzenethiosulphonate group as well as the tetramethylthiourea group occur separately as ligands in such complexes without inducing red colour, the former in $Te(trtu)_2(S_2O_2C_6H_5)_2$,[3] (trtu = trimethylenethiourea), and $Te(etu)_2(S_2O_2C_6H_5)_2$,[4] (etu = ethylenethiourea), and the latter in $Te(tmtu)_2(S_2O_2CH_3)_2$,[1] (tmtu = tetramethylthiourea). The "abnormal" colour of $Te(tmtu)_2(S_2O_2C_6H_5)_2$ would be expected to be associated with the tellurium-ligand bonding. A crystal structure study of this compound has therefore been carried out. It was also thought of interest to record the visible spectrum of the complex, together with the spectra of some of the related, yellow compounds.

EXPERIMENTAL

The crystals of $Te(tmtu)_2(S_2O_2C_6H_5)_2$ were prepared, and unit cell and space group determined by Foss and Johannessen.[1]

For redetermination of unit cell dimensions, 2θ-values of 51 reflections were measured from zero-layer Weissenberg photographs around the a and b axes, using $CuK\alpha_1$ radiation ($\lambda=1.5405$ Å). Sodium chloride powder lines ($a=5.6394$ Å at 18°C) [5] were superimposed on the films for reference.

Intensity data were collected for the $h0l-h3l$, $0kl$, and $1kl$ reflections, using multiple-film, integrating, equi-inclination Weissenberg techniques with (Ni-filtered) $CuK\alpha$ radiation. The crystal rotating about the a axis had the dimensions, given as distances from a common origin to faces: Distance to (101), $(\bar{1}0\bar{1})$, $(10\bar{1})$, and $(\bar{1}01)=0.045$ mm; to $(0\bar{1}1)$ and $(0\bar{1}\bar{1})=0.062$ mm; to $(010)=0.073$ mm. The dimensions of the crystal rotating about the b axis were: Distances to (001) and $(00\bar{1})=0.056$ mm; to (101) and $(\bar{1}0\bar{1})=0.048$ mm; to $(10\bar{1})$ and $(\bar{1}01)=0.049$ mm; to $(0\bar{1}1)$ and $(0\bar{1}\bar{1})=0.069$ mm; to $(010)=0.082$ mm.

Out of 1681 accessible, independent reflections, 1251 had intensities strong enough to be estimated visually by comparison with a scale of timed exposures. The remaining 430 reflections were assigned an intensity equal to the observable limit, and labelled as unobserved reflections.

Estimated corrections for the splitting of α_1 and α_2 at high angles, and absorption, Lorentz, and polarization corrections were applied ($\mu = 120$ cm^{-1}). The absorption correction was based on a modified version of the method described by Busing and Levy,[6] using an $8 \times 8 \times 8$ grid for each of the crystals.

The calculated structure factors were based on the scattering factor curves listed in *International Tables* (Ref. 5, p. 202). Using the $\Delta f'$ and $\Delta f''$ values given by Cromer,[7] the tellurium and sulphur scattering factor curves were corrected for anomalous dispersion, by taking the amplitude of f as the corrected value.

The structure was refined by a least squares, full-matrix program minimizing the function

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

where K is a scale factor. The weight, W, is defined by $W = 1/[(Ka_1)^2 + \sigma^2(F_o)]$, where a_1 is a constant and $\sigma(F_o)$ is the estimated standard deviation of 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.

The calculations were carried out on an IBM 360/50 H computer. Most computer programs were made available by the Weizmann Institute of Science, Rehovoth, Israel, and modified for the IBM computer by Dr. D. Rabinovich. A program calculating weighted least squares planes was written by Mr. Knut Maartmann-Moe, of this Institute; and two other programs, one for Fourier summations and another for extinction corrections, were written by Kjell Åse.

Measurement of reflectance spectra in the

Table 1. Atomic coordinates in fractions of monoclinic cell edges. Isotropic thermal parameters (Å^2) in the form $\exp[-8\pi^2 U(\sin^2\theta/\lambda^2)]$. Standard deviations from the least squares refinement in parentheses.

	x	y	z	U
Te	0	0	0	
S(1)	−0.0190(4)	0.2646(6)	0.0326(3)	
S(2)	0.2636(4)	0.0735(6)	0.0401(3)	
S(3)	0.3617(4)	0.0024(7)	0.1653(3)	
O(1)	0.3346(15)	−0.1367(17)	0.1704(8)	0.084(5)
O(2)	0.5043(13)	0.0442(15)	0.1917(7)	0.072(4)
N(1)	−0.2606(13)	0.2544(18)	0.0575(8)	0.060(4)
N(2)	−0.2492(12)	0.3977(17)	−0.0532(7)	0.047(3)
C(1)	−0.1889(14)	0.3049(21)	0.0075(9)	0.050(4)
C(2)	−0.4136(20)	0.2348(25)	0.0237(11)	0.083(6)
C(3)	−0.1936(17)	0.1834(23)	0.1440(11)	0.072(5)
C(4)	−0.1979(17)	0.4208(22)	−0.1273(10)	0.061(5)
C(5)	−0.3522(20)	0.4936(24)	−0.0453(12)	0.078(6)
C(6)	0.2869(15)	0.0889(21)	0.2346(9)	0.047(4)
C(7)	0.1826(19)	0.0179(22)	0.2588(11)	0.073(5)
C(8)	0.1256(21)	0.0883(27)	0.3162(12)	0.081(6)
C(9)	0.1661(19)	0.2167(25)	0.3400(12)	0.072(6)
C(10)	0.2660(19)	0.2831(26)	0.3145(12)	0.083(6)
C(11)	0.3289(17)	0.2196(23)	0.2615(11)	0.065(5)

Table 2. Anisotropic thermal parameters (Å^2) 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. Standard deviations from the least squares refinement in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Te	387(7)	378(9)	455(6)	55(15)	−5(11)	140(5)
S(1)	445(19)	452(35)	1049(30)	66(40)	−114(31)	207(20)
S(2)	528(20)	693(39)	588(20)	28(39)	−2(28)	201(17)
S(3)	475(19)	546(31)	715(21)	0(53)	50(36)	124(17)

Table 3. Observed and calculated structure factors (× 10). Unobserved reflections are indicated by a minus sign on *F*(O) and included at the threshold values.

H	K	L	F(O)	F(C)
0	0	2	1262	1303
0	0	4	1455	1609
0	0	6	1491	1587
0	0	8	186	165
0	0	10	148	-117
0	0	12	340	315
0	0	14	-100	20
0	0	16	185	167
0	0	18	208	192
0	1	1	1150	1347
0	1	2	-38	-47
0	1	3	248	210
0	1	4	1050	-1109
0	1	5	1222	1257
0	1	6	536	-535
0	1	7	718	761
0	1	8	332	-344
0	1	9	188	190
0	1	10	334	-335
0	1	11	428	455
0	1	12	-95	-58
0	1	13	312	303
0	1	14	-101	-8
0	1	15	75	74
0	1	16	-103	-20
0	1	17	182	154
0	1	18	-76	33
0	1	19	126	138
0	2	0	927	950
0	2	1	658	-741
0	2	2	172	93
0	2	3	291	-238
0	2	4	455	436
0	2	5	380	-432
0	2	6	665	699
0	2	7	137	-129
0	2	8	367	350
0	2	9	238	-223
0	2	10	379	352
0	2	11	-93	4
0	2	12	411	392
0	2	13	108	97
0	2	14	232	181
0	2	15	109	95
0	2	16	203	181
0	2	17	-90	49
0	2	18	204	185
0	3	1	1028	1190
0	3	2	326	285
0	3	3	739	704
0	3	4	404	481
0	3	5	926	984
0	3	6	76	17
0	3	7	675	702
0	3	8	177	188
0	3	9	339	333
0	3	10	-93	17
0	3	11	450	474
0	3	12	-99	33
0	3	13	367	336
0	3	14	-105	-35
0	3	15	176	162
0	3	16	99	66
0	3	17	210	187
0	3	18	-66	43
0	4	0	1524	1704
0	4	1	480	-458
0	4	2	870	875
0	4	3	224	-221
0	4	4	873	905
0	4	5	404	-416
0	4	6	784	842
0	4	7	429	-430
0	4	8	395	410
0	4	9	107	-112
0	4	10	264	260
0	4	11	163	-134
0	4	12	446	408
0	4	13	-104	-8
0	4	14	207	177
0	4	15	111	92
0	4	16	171	136
0	4	17	-78	27
0	4	18	164	147
0	5	1	469	511
0	5	2	331	-323
0	5	3	96	-58
0	5	4	343	-359
0	5	5	417	442
0	5	6	491	-525
0	5	7	556	588
0	5	8	327	-308
0	5	9	223	210
0	5	10	102	-82
0	5	11	490	478
0	5	12	-105	-63
0	5	13	333	287
0	5	14	-107	28
0	5	15	293	243
0	5	16	-85	47
0	5	17	179	161
0	6	0	148	121
0	6	1	305	-261
0	6	2	79	39
0	6	3	134	124
0	6	4	132	130
0	6	5	170	146
0	6	6	644	652
0	6	7	-95	12
0	6	8	439	451
0	6	9	-104	-15
0	6	10	417	423
0	6	11	121	75
0	6	12	532	513
0	6	13	-109	-3
0	6	14	349	282
0	6	15	-88	13
0	6	16	161	148
0	7	1	576	589
0	7	2	-87	-57
0	7	3	172	159
0	7	4	242	253
0	7	5	487	493
0	7	6	188	157
0	7	7	534	572
0	7	8	-103	47
0	7	9	242	238
0	7	10	119	102
0	7	11	404	370
0	7	12	-110	8
0	7	13	343	299
0	7	14	-89	24
0	7	15	147	144
0	8	0	611	599
0	8	1	-94	67
0	8	2	386	387
0	8	3	-95	0
0	8	4	312	342
0	8	5	-99	29
0	8	6	425	398
0	8	7	-104	-41
0	8	8	144	174
0	8	9	-113	25
0	8	10	165	144
0	8	11	-108	-48
0	8	12	314	257
0	8	13	-86	-24
0	8	14	111	121
0	9	1	175	160
0	9	2	-100	-63
0	9	3	-101	78
0	9	4	-103	-29
0	9	5	276	255
0	9	6	-108	-13
0	9	7	344	321
0	9	8	-113	-25
0	9	9	217	210
0	9	10	-104	30
0	9	11	296	276
0	9	12	-80	-7
0	9	13	220	206
0	10	0	340	360
0	10	1	-108	42
0	10	2	167	149
0	10	3	115	112
0	10	4	209	196
0	10	5	-110	89
0	10	6	303	286
0	10	7	126	113
0	10	8	232	217
0	10	9	-94	69
0	10	10	151	140
0	10	11	69	59
0	10	12	168	206
0	11	1	313	299
0	11	2	-105	59
0	11	3	214	200
0	11	4	129	108
0	11	5	249	238
0	11	6	162	128
0	11	7	214	207
0	11	8	-78	57
0	11	9	112	109
0	12	0	311	305
0	12	1	-85	-36
0	12	2	211	203
0	12	3	-81	-27
0	12	4	151	159
0	12	5	-72	-27
0	12	6	155	160
1	0	2	1047	1129
1	0	4	520	661
1	0	6	324	314
1	0	8	460	420
1	0	10	443	429
1	0	12	258	267
1	0	-2	2013	2134
1	0	-4	1342	1473
1	0	-6	827	850
1	0	-8	1182	1293
1	0	-10	519	521
1	0	-12	217	227
1	0	-14	303	289
1	0	-16	92	56
1	0	-18	-97	50
1	1	0	256	198
1	1	1	280	-344
1	1	2	708	665
1	1	3	2188	1935
1	1	4	473	-453
1	1	5	188	237
1	1	6	63	-74
1	1	7	202	199
1	1	8	175	-157
1	1	9	478	491
1	1	10	99	-96
1	1	11	412	403
1	1	12	76	-27
1	1	13	135	126
1	1	-1	1412	1396
1	1	-2	937	948
1	1	-3	1172	1284
1	1	-4	90	103
1	1	-5	447	396
1	1	-6	393	355
1	1	-7	931	989
1	1	-8	204	193
1	1	-9	847	899
1	1	-10	158	139
1	1	-11	209	195
1	1	-12	-106	25
1	1	-13	542	537
1	1	-14	161	144
1	1	-15	331	291
1	1	-16	-119	45
1	1	-17	100	78
1	1	-18	66	24
1	1	-19	124	125
1	2	0	129	-85
1	2	1	718	-707
1	2	2	660	-548
1	2	3	714	-707
1	2	4	665	609
1	2	5	272	246
1	2	6	223	-184
1	2	7	187	169
1	2	8	269	259
1	2	9	74	61
1	2	10	542	521
1	2	11	245	264
1	2	12	368	393
1	2	13	124	112
1	2	14	300	288
1	2	-1	489	-526
1	2	-2	1953	1802
1	2	-3	713	-744
1	2	-4	974	964
1	2	-5	332	-308
1	2	-6	653	703
1	2	-7	157	153
1	2	-8	1353	1386
1	2	-9	-94	-26
1	2	-10	582	609
1	2	-11	176	175
1	2	-12	328	328
1	2	-13	112	116
1	2	-14	418	395
1	2	-15	-119	27
1	2	-16	222	183
1	2	-17	-110	-20
1	2	-18	114	117
1	2	-19	-68	-4
1	3	0	387	-416
1	3	1	625	624
1	3	2	237	-228
1	3	3	1243	1228
1	3	4	446	-430
1	3	5	753	767
1	3	6	377	360
1	3	7	453	455
1	3	8	191	151
1	3	9	495	492
1	3	10	233	226
1	3	11	431	413
1	3	12	193	157
1	3	13	225	211
1	3	14	-121	61
1	3	15	300	244
1	3	-1	1232	1239
1	3	-2	848	-894
1	3	-3	1252	1307
1	3	-4	602	-587
1	3	-5	492	488
1	3	-6	316	-291
1	3	-7	728	710
1	3	-8	92	-72
1	3	-9	633	628
1	3	-10	-104	6
1	3	-11	275	255
1	3	-12	-111	48
1	3	-13	293	292
1	3	-14	-117	100
1	3	-15	289	254
1	3	-16	-118	74
1	3	-17	139	125
1	3	-18	-86	11
1	3	-19	110	119
1	4	0	672	692
1	4	1	349	-348
1	4	2	1195	1209
1	4	3	-75	-86
1	4	4	1030	1112
1	4	5	120	-103
1	4	6	704	711
1	4	7	-98	37
1	4	8	503	511
1	4	9	-108	53
1	4	10	453	474
1	4	11	222	217
1	4	12	176	150
1	4	13	-121	36
1	4	14	201	158
1	4	15	112	74
1	4	-1	583	-590
1	4	-2	779	830
1	4	-3	72	59
1	4	-4	600	608
1	4	-5	233	238
1	4	-6	200	171
1	4	-7	-92	-33
1	4	-8	574	606
1	4	-9	264	273
1	4	-10	280	247
1	4	-11	355	354
1	4	-12	280	296
1	4	-13	117	106
1	4	-14	312	296
1	4	-15	135	106
1	4	-16	241	206
1	4	-17	-98	33
1	4	-18	111	99
1	5	0	356	-351
1	5	1	324	302
1	5	2	268	265
1	5	3	587	614
1	5	4	382	-399
1	5	5	512	547
1	5	6	210	-187
1	5	7	359	377
1	5	8	-110	-54
1	5	9	444	450
1	5	10	-116	-5
1	5	11	395	370
1	5	12	-123	-10
1	5	13	195	163
1	5	14	-118	54
1	5	15	221	196
1	5	-1	539	551
1	5	-2	111	57
1	5	-3	588	592
1	5	-4	267	242
1	5	-5	237	202
1	5	-6	-94	-24
1	5	-7	299	275
1	5	-8	117	97
1	5	-9	502	487
1	5	-10	223	209
1	5	-11	160	135
1	5	-12	-118	-7
1	5	-13	314	278
1	5	-14	123	81
1	5	-15	327	280
1	5	-16	-105	28
1	5	-17	176	167
1	6	0	177	188
1	6	1	-89	18
1	6	2	333	352
1	6	3	-93	-20
1	6	4	731	783
1	6	5	-102	-7
1	6	6	308	297
1	6	7	117	105
1	6	8	393	429
1	6	9	118	66
1	6	10	608	582
1	6	11	-125	13
1	6	12	242	205
1	6	13	135	75
1	6	14	208	180
1	6	15	-89	2
1	6	-1	286	-269
1	6	-2	513	517
1	6	-3	96	-84
1	6	-4	259	278
1	6	-5	203	-205
1	6	-6	212	200
1	6	-7	254	-232
1	6	-8	587	559
1	6	-9	-115	-12
1	6	-10	167	179
1	6	-11	136	-133
1	6	-12	319	302
1	6	-13	-126	3
1	6	-14	369	328
1	6	-15	-109	-8
1	6	-16	218	205
1	6	-17	-67	-4
1	7	0	98	-71
1	7	1	428	515
1	7	2	-100	-26
1	7	3	747	740
1	7	4	-105	51
1	7	5	776	794
1	7	6	176	178
1	7	7	315	354
1	7	8	169	143
1	7	9	488	444
1	7	10	-126	84
1	7	11	333	264
1	7	12	-120	51
1	7	13	107	95
1	7	14	-90	2
1	7	15	153	137
1	7	-1	684	714
1	7	-2	194	-198
1	7	-3	648	617
1	7	-4	-103	-43
1	7	-5	105	102
1	7	-6	-109	-79
1	7	-7	335	324
1	7	-8	-116	9
1	7	-9	275	263
1	7	-10	-123	30
1	7	-11	128	139
1	7	-12	-126	1
1	7	-13	209	172
1	7	-14	-108	7
1	7	-15	207	200
1	7	-16	-70	-5
1	8	0	322	373
1	8	1	131	150
1	8	2	410	417
1	8	3	-109	21
1	8	4	667	696
1	8	5	-113	-120
1	8	6	298	324
1	8	7	-121	-78
1	8	8	240	224
1	8	9	142	-110
1	8	10	350	331
1	8	11	-118	-93
1	8	12	128	104
1	8	13	-88	-58
1	8	-1	130	125
1	8	-2	565	574
1	8	-3	-108	109
1	8	-4	247	259
1	8	-5	177	178
1	8	-6	162	128
1	8	-7	-118	-24
1	8	-8	329	324
1	8	-9	-125	81
1	8	-10	156	133
1	8	-11	-126	-35
1	8	-12	132	120
1	8	-13	-105	-11
1	8	-14	202	195
1	8	-15	-67	-1
1	9	0	113	100
1	9	1	-113	53
1	9	2	-114	-9
1	9	3	348	374
1	9	4	-119	0
1	9	5	330	337
1	9	6	-124	-92
1	9	7	228	194
1	9	8	172	-136
1	9	9	249	247
1	9	10	-110	-75
1	9	11	237	228
1	9	12	-80	-45
1	9	13	79	83
1	9	-1	256	294
1	9	-2	-114	83
1	9	-3	199	207

visible and ultraviolet region were performed on a Zeiss spectrophotometer PMQ II equipped with an integrating sphere. The finely powdered compound was kept in a 0.05 cm quartz cell, and the absorption of light in the cell window was ignored. A sample of MgO, built into the instrument, was used as a white standard of 100 % reflectance. In addition to the tetramethylthiourea complex, the spectra of the corresponding trimethylenethiourea complex and ethylenethiourea complex were measured.

CRYSTAL DATA

The crystals of Te(tmtu)$_2$(S$_2$O$_2$C$_6$H$_5$)$_2$ are monoclinic. Most of them were prisms elongated

Table 3. Continued.

H	K	L	F(O)	F(C)
1	9	-4	175	145
1	9	-5	-119	63
1	9	-6	-122	43
1	9	-7	154	145
1	9	-8	-127	-46
1	9	-9	310	289
1	9	-10	-122	-32
1	9	-11	136	117
1	9	-12	-98	-36
1	9	-13	196	192
1	9	-14	-57	-67
1	10	0	156	171
1	10	1	-123	53
1	10	2	144	133
1	10	3	-124	86
1	10	4	228	212
1	10	5	132	70
1	10	6	168	151
1	10	7	-118	48
1	10	8	189	170
1	10	9	-98	55
1	10	10	222	234
1	10	11	-65	-11
1	10	-1	-122	28
1	10	-2	293	256
1	10	-3	-123	-18
1	10	-4	261	261
1	10	-5	-125	-32
1	10	-6	242	248
1	10	-7	-123	-65
1	10	-8	296	267
1	10	-9	-111	-51
1	10	-10	187	206
1	10	-11	85	-90
1	10	-12	100	109
1	11	0	-119	10
1	11	1	252	235
1	11	2	-117	92
1	11	3	330	331
1	11	4	-112	59
1	11	5	237	231
1	11	6	-101	30
1	11	7	121	132
1	11	8	-79	9
1	11	9	137	152
1	11	-1	313	317
1	11	-2	-119	43
1	11	-3	277	266
1	11	-4	-116	15
1	11	-5	195	202
1	11	-6	-108	-27
1	11	-7	203	201
1	11	-8	-93	-54
1	11	-9	151	158
1	11	-10	-62	-14
1	12	1	-94	-15
1	12	2	134	132
1	12	3	-88	-23
1	12	4	191	201
1	12	5	-73	-63
1	12	6	72	98
1	12	-1	-95	8
1	12	-2	236	246
1	12	-3	-92	27
1	12	-4	150	155
1	12	-5	-82	45
1	12	-6	128	137
1	12	-7	-60	30
1	0	0	817	779
1	0	14	306	311
1	0	16	368	338
1	0	18	150	176
2	0	0	714	-588
2	0	2	1954	1844
2	0	4	238	221
2	0	6	917	537
2	0	8	1001	585
2	0	10	567	556
2	0	12	331	333
2	0	14	426	387
2	0	16	113	89
2	0	18	-30	70
2	0	-2	135	-81
2	0	-4	1433	1514
2	0	-6	605	684
2	0	-8	148	125
2	0	-10	1211	1249
2	0	-12	462	460
2	0	-14	233	230
2	0	-16	346	315
2	0	-18	165	139
3	0	0	1367	1484
3	0	2	1209	1187
3	0	4	1395	1452
3	0	6	1005	1044
3	0	8	414	431
3	0	10	351	376
3	0	12	357	330
3	0	14	-89	33
3	0	16	-65	36
3	0	-2	1109	1165
3	0	-4	541	469
3	0	-6	274	251
3	0	-8	104	120
3	0	-10	220	-249
3	0	-12	350	355
3	0	-14	231	207
3	0	-16	184	185
3	0	-18	321	285
3	0	-20	159	183
4	0	0	1278	1302
4	0	2	1105	1126
4	0	4	1237	1263
4	0	6	195	186
4	0	8	176	160
4	0	10	275	277
4	0	12	-91	0
4	0	14	-79	70
4	0	16	138	169
4	0	-2	1877	2003
4	0	-4	1052	1086
4	0	-6	428	430
4	0	-8	593	659
4	0	-10	268	279
4	0	-12	140	-125
4	0	-14	330	341
4	0	-16	215	213
4	0	-18	100	104
4	0	-20	140	177
5	0	0	810	876
5	0	2	1192	1145
5	0	4	246	230
5	0	6	492	531
5	0	8	610	595
5	0	10	100	105
5	0	12	303	274
5	0	14	246	235
5	0	-2	830	843
5	0	-4	946	939
5	0	-6	868	943
5	0	-8	702	698
5	0	-10	655	646
5	0	-12	373	332
5	0	-14	274	285
5	0	-16	180	162
5	0	-18	152	145
5	0	-20	-53	52
6	0	0	882	858
6	0	2	82	34
6	0	4	681	697
6	0	6	680	721
6	0	8	349	382
6	0	10	487	440
6	0	12	299	295
6	0	-2	323	353
6	0	-4	70	37
6	0	-6	563	648
6	0	-8	583	640
6	0	-10	136	139
6	0	-12	588	627
6	0	-14	294	288
6	0	-16	214	217
6	0	-18	205	202
6	0	-20	139	164
7	0	0	465	460
7	0	2	850	860
7	0	4	780	801
7	0	6	360	363
7	0	8	446	420
7	0	10	266	261
7	0	12	60	57
7	0	-2	719	679
7	0	-4	560	578
7	0	-6	87	-65
7	0	-8	169	157
7	0	-10	160	107
7	0	-12	145	156
7	0	-14	142	144
7	0	-16	282	251
7	0	-18	182	195
8	0	0	786	756
8	0	2	641	685
8	0	4	292	290
8	0	6	405	369
8	0	8	156	143
8	0	10	-58	39
8	0	-2	373	411
8	0	-4	482	546
8	0	-6	521	524
8	0	-8	290	-277
8	0	-10	254	275
8	0	-12	97	80
8	0	-14	-92	28
8	0	-16	140	130
8	0	-18	166	173
9	0	0	229	236
9	0	2	216	182
9	0	4	305	290
9	0	6	207	204
9	0	8	110	107
9	0	-2	475	476
9	0	-4	254	249
9	0	-6	364	379
9	0	-8	557	547
9	0	-10	305	269
9	0	-12	261	245
9	0	-14	353	303
9	0	-16	132	120
9	0	-18	74	89
10	0	0	202	164
10	0	2	350	330
10	0	4	326	295
10	0	6	215	235
10	0	-2	123	123
10	0	-4	279	231
10	0	-6	89	39
10	0	-8	241	258
10	0	-10	320	287
10	0	-12	184	195
10	0	-14	220	232
10	0	-16	275	283
11	0	0	452	432
11	0	2	316	295
11	0	4	215	247
11	0	-2	221	206
11	0	-4	154	127
11	0	-6	138	163
11	0	-8	-89	8
11	0	-10	-87	19
11	0	-12	135	128
11	0	-14	116	135
11	0	-16	93	124
12	0	0	214	204
12	0	-2	328	313
12	0	-4	191	188
12	0	-6	166	146
12	0	-8	192	168
12	0	-10	89	89
12	0	-12	73	73
12	0	-14	85	108
1	1	14	83	73
1	1	15	335	301
1	1	16	-71	49
1	1	17	219	224
1	1	18	-46	28
2	1	0	48	54
2	1	1	1184	1130
2	1	2	296	-195
2	1	3	708	641
2	1	4	383	329
2	1	5	191	160
2	1	6	339	-331
2	1	7	704	676
2	1	8	-58	37
2	1	9	614	619
2	1	10	-65	-45
2	1	11	344	336
2	1	12	123	-124
2	1	13	401	380
2	1	14	-75	-4
2	1	15	380	328
2	1	16	-64	6
2	1	17	105	130
2	1	-1	333	290
2	1	-2	309	408
2	1	-3	521	537
2	1	-4	-33	40
2	1	-5	1246	1285
2	1	-6	68	8
2	1	-7	431	408
2	1	-8	392	406
2	1	-9	594	642
2	1	-10	-56	-1
2	1	-11	702	769
2	1	-12	85	89
2	1	-13	343	326
2	1	-14	78	46
2	1	-15	409	410
2	1	-16	-76	19
2	1	-17	371	316
2	1	-18	-66	4
2	1	-19	114	126
3	1	0	245	202
3	1	1	973	908
3	1	2	-42	-56
3	1	3	357	314
3	1	4	116	-97
3	1	5	1214	1186
3	1	6	102	-105
3	1	7	649	685
3	1	8	150	-146
3	1	9	424	414
3	1	10	123	-107
3	1	11	439	434
3	1	12	-73	-73
3	1	13	323	298
3	1	14	97	-73
3	1	15	69	80
3	1	16	-53	-27
3	1	-1	1267	1282
3	1	-2	251	167
3	1	-3	240	268
3	1	-4	843	846
3	1	-5	213	261
3	1	-6	370	398
3	1	-7	922	914
3	1	-8	252	271
3	1	-9	171	174
3	1	-10	153	159
3	1	-11	382	335
3	1	-12	-64	-34
3	1	-13	467	498
3	1	-14	126	-154
3	1	-15	191	185
3	1	-16	126	-115
3	1	-17	322	310
3	1	-18	-69	-85
3	1	-19	221	230
4	1	0	252	-171
4	1	1	616	539
4	1	2	339	-257
4	1	3	789	824
4	1	4	363	-305
4	1	5	784	744
4	1	6	251	-246
4	1	7	157	150
4	1	8	331	-321
4	1	9	429	388
4	1	10	140	-110
4	1	11	194	189
4	1	12	-75	-23
4	1	13	81	85
4	1	14	-65	-29
4	1	15	176	171
4	1	-1	1239	1163
4	1	-2	350	406
4	1	-3	1232	1206
4	1	-4	-43	75
4	1	-5	646	681
4	1	-6	467	496
4	1	-7	432	477
4	1	-8	-52	30
4	1	-9	735	729
4	1	-10	101	80
4	1	-11	287	267
4	1	-12	-65	8
4	1	-13	124	126
4	1	-14	-70	-28
4	1	-15	285	256
4	1	-16	94	-106
4	1	-17	154	125
4	1	-18	-70	-17
4	1	-19	118	110
5	1	0	258	208
5	1	1	754	749
5	1	2	61	29
5	1	3	344	302
5	1	4	-60	4
5	1	5	70	24
5	1	6	171	-175
5	1	7	515	493
5	1	8	128	-140
5	1	9	178	158
5	1	10	108	-111
5	1	11	187	171
5	1	12	-72	-52
5	1	13	306	283
5	1	14	-51	-22
5	1	-1	108	76
5	1	-2	356	317
5	1	-3	834	802
5	1	-4	226	213
5	1	-5	965	1034
5	1	-6	65	69
5	1	-7	583	614
5	1	-8	167	218
5	1	-9	553	601
5	1	-10	-61	-11
5	1	-11	741	784
5	1	-12	146	95
5	1	-13	273	300
5	1	-14	94	89
5	1	-15	268	249
5	1	-16	98	50
5	1	-17	201	173
5	1	-18	-69	24
5	1	-19	109	103
6	1	0	388	406
6	1	1	337	350
6	1	2	175	164
6	1	3	217	231
6	1	4	65	75
6	1	5	606	618
6	1	6	-68	45
6	1	7	301	298
6	1	8	-73	2
6	1	9	292	263
6	1	10	-74	-53
6	1	11	420	368
6	1	12	70	-63
6	1	13	126	149
6	1	-1	520	514
6	1	-2	104	104
6	1	-3	89	44
6	1	-4	234	221
6	1	-5	309	319
6	1	-6	177	192
6	1	-7	590	616
6	1	-8	88	61
6	1	-9	278	260
6	1	-10	-63	-20
6	1	-11	451	476
6	1	-12	-68	25
6	1	-13	521	548
6	1	-14	88	-97
6	1	-15	409	392
6	1	-16	-76	40
6	1	-17	237	232
6	1	-18	-66	-19
6	1	-19	225	226
7	1	0	198	207
7	1	1	460	467
7	1	2	107	88
7	1	3	804	785
7	1	4	114	-102
7	1	5	266	248
7	1	6	-72	-14
7	1	7	306	281
7	1	8	95	-61
7	1	9	375	346
7	1	10	-65	-60
7	1	11	99	104
7	1	12	-41	-35
7	1	-1	479	443
7	1	-2	248	250
7	1	-3	791	758
7	1	-4	362	330
7	1	-5	334	340
7	1	-6	426	454
7	1	-7	163	170
7	1	-8	167	139
7	1	-9	487	512
7	1	-10	210	193
7	1	-11	-68	36
7	1	-12	78	-78
7	1	-13	256	242
7	1	-14	-74	-63
7	1	-15	314	280
7	1	-16	93	-98
7	1	-17	262	233
7	1	-18	-62	-50
7	1	-19	160	166
8	1	0	-68	54
8	1	1	515	533
8	1	2	-70	73
8	1	3	182	186
8	1	4	-73	-3
8	1	5	201	187
8	1	6	84	-87
8	1	7	307	286
8	1	8	-67	-45
8	1	9	80	77
8	1	10	-47	-35
8	1	11	-23	100
8	1	-1	586	529
8	1	-2	272	250
8	1	-3	375	375
8	1	-4	254	276
8	1	-5	780	805
8	1	-6	133	103
8	1	-7	459	461
8	1	-8	136	130
8	1	-9	371	396
8	1	-10	-69	56
8	1	-11	393	401
8	1	-12	-72	-52
8	1	-13	194	166
8	1	-14	-76	-28
8	1	-15	89	91
8	1	-16	81	-65
8	1	-17	174	158
8	1	-18	-56	-51
8	1	-19	87	88
9	1	0	-72	60
9	1	1	90	75
9	1	2	-75	71
9	1	3	198	168
9	1	4	-75	47
9	1	5	292	278
9	1	6	-66	-23
9	1	7	117	103
9	1	8	-48	-45
9	1	9	-32	137
9	1	-1	332	348
9	1	-2	-70	66
9	1	-3	398	387
9	1	-4	-69	92
9	1	-5	138	128
9	1	-6	142	-107
9	1	-7	563	594
9	1	-8	70	-31
9	1	-9	444	433
9	1	-10	89	-81
9	1	-11	360	341
9	1	-12	-76	-5
9	1	-13	454	390
9	1	-14	-76	-27
9	1	-15	276	245
9	1	-16	-66	22
9	1	-17	106	113
9	1	-18	-47	-5
10	1	0	173	141
10	1	1	279	245
10	1	2	-72	71
10	1	3	272	275
10	1	4	-62	59
10	1	5	166	166
10	1	6	-46	7
10	1	-1	119	93
10	1	-2	188	157
10	1	-3	289	297
10	1	-4	-74	57
10	1	-5	212	204
10	1	-6	141	107
10	1	-7	123	105
10	1	-8	-74	-7
10	1	-9	249	249
10	1	-10	-75	-59
10	1	-11	339	295
10	1	-12	-75	-63

in the b axis direction, bounded by {001}, {101}, and {$10\overline{1}$}, and terminated by {011}. Based on 51 measured 2θ-values, a least squares procedure gave the following unit cell dimensions: $a = 10.254(5)$ Å; $b = 10.029(8)$ Å; $c = 15.929(7)$ Å; $\beta = 110.35(5)°$. Other pertinent data are:

Table 3. Continued.

H	K	L	F(O)	F(C)
10	1	-13	210	157
10	1	-14	-69	-51
10	1	-15	266	265
10	1	-16	-55	-59
10	1	-17	131	218
11	1	0	109	108
11	1	1	296	290
11	1	2	-56	46
11	1	3	144	148
11	1	4	-39	3
11	1	-1	296	289
11	1	-2	133	128
11	1	-3	182	148
11	1	-4	171	127
11	1	-5	333	319
11	1	-6	160	129
11	1	-7	253	229
11	1	-8	94	78
11	1	-9	73	76
11	1	-10	0	5
11	1	-11	172	158
11	1	-12	64	-70
11	1	-13	179	175
11	1	-14	60	-44
11	1	-15	74	79
11	1	-16	74	-67
12	1	0	75	69
12	1	1	67	93
12	1	-1	264	249
12	1	-2	86	72
12	1	-3	228	250
12	1	-4	97	83
12	1	-5	232	211
12	1	-6	-61	54
12	1	-7	297	292
12	1	-8	-50	44
12	1	-9	256	252
12	1	-10	-57	1
12	1	-11	140	146
12	1	-12	-50	18
12	1	-13	119	135
13	1	-5	134	187
13	1	-6	-36	-14
13	1	-7	105	141
13	1	-8	-37	-53
13	1	-9	208	230
0	2	19	-37	47
1	2	15	-94	40
1	2	16	346	327
1	2	17	-73	-13
1	2	18	103	145
2	2	0	446	475
2	2	1	209	202
2	2	2	446	448
2	2	3	317	313
2	2	4	148	144
2	2	5	449	445
2	2	6	837	836
2	2	7	-69	26
2	2	8	532	546
2	2	9	249	236
2	2	10	451	486
2	2	11	-85	-42
2	2	12	365	416
2	2	13	-93	-66
2	2	14	412	418
2	2	15	-90	-71
2	2	16	157	171
2	2	17	-60	-59
2	2	-1	327	-318
2	2	-2	316	-288
2	2	-3	509	-532
2	2	-4	502	540
2	2	-5	531	-583
2	2	-6	481	491
2	2	-7	194	-202
2	2	-8	336	318
2	2	-9	473	-450
2	2	-10	858	865
2	2	-11	-77	-58
2	2	-12	584	613
2	2	-13	-85	69
2	2	-14	368	379
2	2	-15	-93	46
2	2	-16	461	448
2	2	-17	-92	57
2	2	-18	187	189
2	2	-19	-63	41
3	2	0	639	640
3	2	1	929	902
3	2	2	846	809
3	2	3	246	216
3	2	4	443	409
3	2	5	180	-165
3	2	6	923	933
3	2	7	-74	11
3	2	8	373	344
3	2	9	154	-157
3	2	10	333	319
3	2	11	108	-121
3	2	12	368	367
3	2	13	106	-82
3	2	14	156	155
3	2	15	-79	-45
3	2	16	77	103
3	2	17	-21	26
3	2	-1	160	177
3	2	-2	668	630
3	2	-3	115	134
3	2	-4	116	30
3	2	-5	688	671
3	2	-6	808	862
3	2	-7	406	-407
3	2	-8	492	602
3	2	-9	109	-91
3	2	-10	326	354
3	2	-11	189	-178
3	2	-12	593	638
3	2	-13	172	-159
3	2	-14	502	525
3	2	-15	-93	-56
3	2	-16	220	205
3	2	-17	-94	-2
3	2	-18	279	274
3	2	-19	-68	-6
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4	2	0	283	240
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4	2	2	425	448
4	2	3	250	-243
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4	2	5	249	-239
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4	2	7	90	-79
4	2	8	435	440
4	2	9	110	-49
4	2	10	366	366
4	2	11	-92	57
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4	2	13	-90	46
4	2	14	177	179
4	2	15	-63	73
4	2	16	-30	196
4	2	-1	170	127
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4	2	-3	135	129
4	2	-4	815	895
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4	2	-7	270	291
4	2	-8	773	736
4	2	-9	170	166
4	2	-10	580	627
4	2	-11	121	117
4	2	-12	219	226
4	2	-13	-85	-65
4	2	-14	359	344
4	2	-15	-93	-36
4	2	-16	186	156
4	2	-17	-94	-41
4	2	-18	135	119
4	2	-19	-70	-36
4	2	-20	71	119
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5	2	4	159	-133
5	2	5	79	79
5	2	6	104	51
5	2	7	155	131
5	2	8	363	346
5	2	9	131	131
5	2	10	136	117
5	2	11	136	107
5	2	12	242	246
5	2	13	-76	70
5	2	14	184	242
5	2	15	-12	25
5	2	-1	473	-454
5	2	-2	159	143
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5	2	-4	789	874
5	2	-5	493	-516
5	2	-6	718	745
5	2	-7	-67	-36
5	2	-8	541	588
5	2	-9	243	234
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6	2	6	376	363
6	2	7	94	93
6	2	8	-92	61
6	2	9	94	41
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6	2	-3	237	-199
6	2	-4	276	226
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6	2	-7	388	-373
6	2	-8	640	642
6	2	-9	202	-199
6	2	-10	460	490
6	2	-11	191	-161
6	2	-12	552	523
6	2	-13	-88	-9
6	2	-14	427	407
6	2	-15	-95	-24
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6	2	-19	-66	12
6	2	-20	92	152
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7	2	5	-88	-69
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7	2	7	-94	-86
7	2	8	336	313
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7	2	-9	170	-167
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7	2	-11	210	-174
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7	2	-17	-87	-58
7	2	-18	163	165
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8	2	5	145	-136
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8	2	7	105	-75
8	2	8	132	120
8	2	9	-70	7
8	2	10	-55	64
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8	2	-2	331	337
8	2	-3	-83	32
8	2	-4	582	551
8	2	-5	-83	16
8	2	-6	822	839
8	2	-7	271	235
8	2	-8	391	408
8	2	-9	-85	23
8	2	-10	458	431
8	2	-11	89	78
8	2	-12	449	435
8	2	-13	-94	20
8	2	-14	216	185
8	2	-15	-94	3
8	2	-16	181	160
8	2	-17	-80	-56
8	2	-18	158	161
8	2	-19	-49	-14
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9	2	1	-92	-51
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9	2	3	-94	-49
9	2	4	151	122
9	2	5	-88	37
9	2	6	120	96
9	2	7	-70	56
9	2	8	-57	32
9	2	9	-29	71
9	2	-1	219	-184
9	2	-2	513	501
9	2	-3	191	-149
9	2	-4	287	291
9	2	-5	119	-111
9	2	-6	546	592
9	2	-7	88	-81
9	2	-8	567	570
9	2	-9	-90	31
9	2	-10	314	324
9	2	-11	132	87
9	2	-12	318	314
9	2	-13	-95	43
9	2	-14	349	311
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9	2	-16	98	103
9	2	-17	69	65
9	2	-18	100	117
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10	2	1	-91	29
10	2	2	207	213
10	2	3	83	63
10	2	4	125	105
10	2	5	93	77
10	2	6	-54	63
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10	2	-3	-93	-75
10	2	-4	367	376
10	2	-5	-93	-80
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10	2	-7	152	-138
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10	2	-12	302	271
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10	2	-14	273	241
10	2	-15	-76	-28
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10	2	-17	-53	49
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11	2	3	-57	-8
11	2	4	72	108
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11	2	-2	178	171
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11	2	-4	282	254
11	2	-5	-91	21
11	2	-6	307	287
11	2	-7	-91	-22
11	2	-8	276	246
11	2	-9	-90	-16
11	2	-10	203	191
11	2	-11	-84	-53
11	2	-12	293	259
11	2	-13	118	-79
11	2	-14	140	135
11	2	-15	-58	-25
11	2	-16	80	127
12	2	0	106	115
12	2	1	-35	-58
12	2	-1	71	-85
12	2	-2	287	336
12	2	-3	-68	-27
12	2	-4	235	253
12	2	-5	-72	-3
12	2	-6	278	277
12	2	-7	-74	29
12	2	-8	327	354
12	2	-9	-71	15
12	2	-10	211	231
12	2	-11	-65	36
12	2	-12	117	139
1	3	16	-214	40
1	3	17	185	175
2	3	0	101	-114
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2	3	2	261	194
2	3	3	534	521
2	3	4	182	166
2	3	5	429	391
2	3	6	299	338
2	3	7	698	696
2	3	8	305	304
2	3	9	398	410
2	3	10	-213	179
2	3	11	313	277
2	3	12	-231	34
2	3	13	342	308
2	3	14	-241	76
2	3	15	247	227
2	3	-1	792	736
2	3	-2	208	165
2	3	-3	305	353
2	3	-4	437	-436
2	3	-5	1048	1183
2	3	-6	489	-529
2	3	-7	300	325
2	3	-8	236	-272
2	3	-9	518	555
2	3	-10	349	-372
2	3	-11	450	494
2	3	-12	-212	-170
2	3	-13	221	189
2	3	-14	-231	-84
2	3	-15	272	216
2	3	-16	-246	47
2	3	-17	256	212
2	3	-18	-195	64
3	3	0	-126	32
3	3	1	720	699
3	3	2	232	190
3	3	3	577	537
3	3	4	235	211
3	3	5	706	682
3	3	6	184	30
3	3	7	517	546
3	3	8	-205	120
3	3	9	311	313
3	3	10	-221	-143
3	3	11	294	304
3	3	12	-241	10
3	3	13	270	227
3	3	-1	708	740
3	3	-2	-121	-8
3	3	-3	435	476
3	3	-4	-127	47
3	3	-5	338	372
3	3	-6	441	-446
3	3	-7	676	693
3	3	-8	275	-268
3	3	-9	399	415
3	3	-10	-190	-108
3	3	-11	284	262
3	3	-12	224	-193
3	3	-13	443	483
3	3	-14	-231	-55
3	3	-15	243	205
3	3	-16	-247	-78
3	3	-17	236	181
3	3	-18	-203	34
3	3	-19	179	172
4	3	0	248	-214
4	3	1	848	838
4	3	2	237	-194
4	3	3	916	882
4	3	4	286	238
4	3	5	605	583
4	3	6	-200	-54
4	3	7	209	210
4	3	9	459	456
4	3	10	-232	73
4	3	11	241	184
4	3	12	-239	19
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4	3	-2	197	-164
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4	3	-6	165	131
4	3	-7	445	464
4	3	-8	-174	-6
4	3	-9	458	462
4	3	-10	-195	93
4	3	-11	316	318
4	3	-12	251	262
4	3	-13	222	195
4	3	-14	-232	24
4	3	-15	356	333
4	3	-16	-248	-13
4	3	-17	236	156
4	3	-18	-207	-26
4	3	-19	165	154
5	3	0	486	-455
5	3	1	741	737
5	3	2	-183	-91
5	3	3	463	465
5	3	4	-198	-52
5	3	5	281	310
5	3	6	213	198
5	3	7	440	461
5	3	8	-225	120
5	3	9	263	248
5	3	10	270	256
5	3	11	237	202
5	3	12	265	88
5	3	-1	647	592
5	3	-2	653	-596
5	3	-3	756	720
5	3	-4	551	-472
5	3	-5	863	889
5	3	-6	366	-358
5	3	-7	641	657
5	3	-8	195	-181
5	3	-9	373	395
5	3	-10	-201	-69
5	3	-11	539	565
5	3	-12	-218	66
5	3	-13	263	280
5	3	-14	-234	110
5	3	-15	245	170
5	3	-16	-246	115
5	3	-17	273	223
5	3	-18	-204	5
5	3	-19	164	141
6	3	0	-192	17
6	3	1	334	322
6	3	2	-202	-32
6	3	3	396	386
6	3	4	-213	82
6	3	5	621	635
6	3	6	-223	42
6	3	7	327	316
6	3	8	-239	105
6	3	9	269	236
6	3	10	-231	55
6	3	11	318	325
6	3	-1	820	776
6	3	-2	186	-189
6	3	-3	312	343
6	3	-4	224	-250

$V = 1535.9(17)$ Å^3; $M = 738.52$; $F(000) = 748$; $Z = 2$. Systematic absences are $h0l$ when l is odd, $0k0$ when k is odd. The uniquely determined space group is $P2_1/c$ (No. 14).

STRUCTURE DETERMINATION

The structure was solved through Fourier synthesis. The tellurium atom, which is located

Table 3. Continued.

H	K	L	F(O)	F(C)
6	3	-5	451	417
6	3	-6	305	-285
6	3	-7	582	596
6	3	-8	333	-279
6	3	-9	445	445
6	3	-10	305	-264
6	3	-11	314	315
6	3	-12	235	-177
6	3	-13	397	390
6	3	-14	-239	-47
6	3	-15	275	240
6	3	-16	-243	51
6	3	-17	225	156
6	3	-18	-196	55
6	3	-19	155	154
7	3	0	-210	54
7	3	1	442	457
7	3	2	-217	-74
7	3	3	597	594
7	3	4	-225	-36
7	3	5	231	247
7	3	6	-239	11
7	3	7	307	297
7	3	8	-236	-14
7	3	9	278	277
7	3	10	-191	-55
7	3	-1	320	326
7	3	-2	-204	-75
7	3	-3	634	616
7	3	-4	-201	-108
7	3	-5	310	312
7	3	-6	-203	-115
7	3	-7	205	174
7	3	-8	-208	-98
7	3	-9	418	419
7	3	-10	-217	-67
7	3	-11	222	193
7	3	-12	-228	-132
7	3	-13	237	162
7	3	-14	-244	-67
7	3	-15	300	263
7	3	-16	-235	-80
7	3	-17	213	168
8	3	0	352	-333
8	3	1	508	504
8	3	2	-230	-195
8	3	3	-236	190
8	3	4	-241	-128
8	3	5	242	228
8	3	6	-236	20
8	3	7	304	278
8	3	8	-198	-18
8	3	-1	456	440
8	3	-2	218	-204
8	3	-3	254	205
8	3	-4	352	-303
8	3	-5	636	567
8	3	-6	-216	-50
8	3	-7	383	351
8	3	-8	-220	21
8	3	-9	222	160
8	3	-10	-226	174
8	3	-11	318	316
8	3	-12	-239	170
8	3	-13	244	212
8	3	-14	-244	83
8	3	-15	-237	103
8	3	-16	-219	37
8	3	-17	216	180
9	3	0	237	-187
9	3	1	-241	174
9	3	2	-242	-63
9	3	3	268	265
9	3	4	-232	-22
9	3	5	315	282
9	3	6	-194	51
9	3	-1	405	377
9	3	-2	337	-311
9	3	-3	396	362
9	3	-4	-228	-122
9	3	-5	227	189
9	3	-6	241	-223
9	3	-7	437	397
9	3	-8	-232	-114
9	3	-9	311	314
9	3	-10	-239	29
9	3	-11	243	159
9	3	-12	-244	66
9	3	-13	296	287
9	3	-14	-233	88
9	3	-15	216	179
10	3	0	-235	-25
10	3	1	302	289
10	3	2	-217	-15
10	3	3	294	281
10	3	4	-181	29
10	3	-1	-239	130
10	3	-2	-241	-104
10	3	-3	410	367
10	3	-4	-241	-71
10	3	-5	352	305
10	3	-6	242	-137
10	3	-7	242	151
10	3	-8	242	-117
10	3	-9	353	285
10	3	-10	256	-171
10	3	-11	336	295
10	3	-12	-231	-64
10	3	-15	233	218
10	3	-14	-203	-26
10	3	-13	-219	109
11	3	0	-191	-93
11	3	1	207	251
11	3	-1	248	268
11	3	-2	-212	-19
11	3	-3	219	153
11	3	-4	-224	-63
11	3	-5	265	239
11	3	-6	-227	9
11	3	-7	278	251
11	3	-8	-225	30
11	3	-9	-221	120
11	3	-10	-215	21
11	3	-11	230	197
11	3	-12	-195	-31
11	3	-13	190	186
12	3	-1	167	225
12	3	-2	-148	-118
12	3	-3	179	203
12	3	-4	168	-133
12	3	-5	-173	102
12	3	-6	-176	-39
12	3	-7	177	178
12	3	-8	-175	31
12	3	-9	190	186
12	3	-10	-162	76

in a centre of symmetry, does not contribute to reflections with $k+l$ odd. With phases based on the tellurium contributions alone, the first three-dimensional Fourier map was calculated for reflections with $k+l$ even. The map had extra symmetry because of the omission of the $k+l$ odd reflections. A set of coordinates for one sulphur atom was picked out arbitrarily among six peaks. On inclusion of some $k+l$ odd reflections, subsequent maps permitted the coordinates of the two remaining sulphur atoms to be determined. It turned out to be difficult to locate the light atoms from three-dimensional Fourier maps, probably because of an insufficient amount of data. It was, however, possible to determine the light atom coordinates from two-dimensional Fourier maps along the a and b axes.

Three-dimensional least squares refinement on scale factors, positional parameters and individual isotropic thermal parameters resulted in an R value of 0.096. Further refinement, where scale factors were not varied, and anisotropic thermal parameters for the tellurium and sulphur atoms were introduced, lowered the R value to 0.083.

Extinction corrections were then carried out, using the expression given by Zachariasen.[8] The absorption term in this expression was set equal to one. With observed intensities on an absolute scale, the value of the extinction parameter, C, was found to be 3.12×10^{-6} for the crystal rotating about the a axis, and 0.76×10^{-6} for the crystal rotating about the b axis.

After introduction of an overall scale factor, the refinement was continued until to parameter shift was greater than 0.18 times the standard deviation. The value of the constant a_1 in the weighting scheme was kept equal to one during the last refinement cycles. The final value of R, including non-observed reflections when $K|F_c|$ exceeded the observable limit, was 0.075. A three-dimensional difference Fourier summation after the last cycle showed no peaks higher than 0.8 e/Å^3.

The final atomic coordinates and isotropic thermal parameters are listed in Table 1, and the final anisotropic thermal parameters for the tellurium and sulphur atoms are listed in Table 2. Observed and calculated structure factors are listed in Table 3.

RESULTS

The reflectance spectrum of the complex of tellurium dibenzenethiosulphonate with tetramethylthiourea, $Te(tmtu)_2(S_2O_2C_6H_5)_2$, is shown in Fig. 1. The spectra of the corresponding trimethylenethiourea complex, $Te(trtu)_2(S_2O_2C_6H_5)_2$, and the ethylenethiourea complex, $Te(etu)_2(S_2O_2C_6H_5)_2$, have been included in Fig. 1 for comparison.

Bond lengths and angles are listed in Table 4. The standard deviations are calculated from those of Table 2, without regard to coordinate covariances and standard deviations in unit cell dimensions. Fig. 2 is a drawing of the molecule, with selected bond lengths and angles.

The tellurium atom is bonded to two tetramethylthiourea sulphur atoms and two benzenethiosulphonate sulphur atoms. With the tellu-

Table 4. Bond lengths (Å) and angles (°). Standard deviations in parentheses.

TeS_4 coordination group			
Te–S(1)	=2.724(6)	∠S(1)–Te–S(2)	=79.51(15)
Te–S(2)	=2.657(4)		
Benzenethiosulphonate group			
S(2)–S(3)	=2.025(6)	∠Te–S(2)–S(3)	=104.7(3)
S(3)–O(1)	=1.430(18)	∠S(2)–S(3)–O(1)	=111.1(6)
S(3)–O(2)	=1.436(13)	∠S(2)–S(3)–O(2)	=107.0(6)
S(3)–C(6)	=1.776(19)	∠S(2)–S(3)–C(6)	=105.5(6)
C(6)–C(7)	=1.444(28)	∠O(1)–S(3)–O(2)	=117.8(9)
C(7)–C(8)	=1.430(32)	∠O(1)–S(3)–C(6)	=107.8(10)
C(8)–C(9)	=1.366(35)	∠O(2)–S(3)–C(6)	=106.9(8)
C(9)–C(10)	=1.395(32)	∠S(3)–C(6)–C(7)	=116.4(15)
C(10)–C(11)	=1.383(31)	∠S(3)–C(6)–C(11)	=119.6(14)
C(11)–C(6)	=1.399(30)	∠C(11)–C(6)–C(7)	=124.0(17)
		∠C(6)–C(7)–C(8)	=115.6(19)
		∠C(7)–C(8)–C(9)	=119.4(22)
		∠C(8)–C(9)–C(10)	=123.5(22)
		∠C(9)–C(10)–C(11)	=120.2(22)
		∠C(10)–C(11)–C(6)	=117.3(19)
Tetramethylthiourea group			
S(1)–C(1)	=1.696(16)	∠Te–S(1)–C(1)	=108.8(8)
C(1)–N(1)	=1.356(23)	∠S(1)–C(1)–N(1)	=119.7(12)
C(1)–N(2)	=1.329(22)	∠S(1)–C(1)–N(2)	=121.2(13)
N(1)–C(2)	=1.485(23)	∠C(1)–N(1)–C(2)	=123.8(12)
N(1)–C(3)	=1.489(22)	∠C(1)–N(1)–C(3)	=123.5(13)
N(2)–C(4)	=1.468(22)	∠C(1)–N(2)–C(4)	=119.3(15)
N(2)–C(5)	=1.466(27)	∠C(1)–N(2)–C(5)	=124.3(14)
		∠C(2)–N(1)–C(3)	=111.2(15)
		∠C(4)–N(2)–C(5)	=115.8(15)
		∠N(1)–C(1)–N(2)	=118.6(14)

rium atom in a centre of symmetry, the TeS_4 coordination is *trans* square-planar. There is a marked difference between the Te–S (benzenethiosulphonate) bond length and the Te–S-(tetramethylthiourea) bond length, the latter being the longer. As is seen in Table 5, a similar tendency has been observed for the corresponding trimethylenethiourea complex and the ethylenethiourea complex. The Te–S bond lengths do not differ much from the average values, 2.68 Å, found for Te–S bond lengths in *trans* square-planar complex of divalent tellurium.[9] There is, however, a striking difference between the present complex and the other complexes of Table 5, inasmuch as the S–Te–S angle in the former differs with about 10° from the corresponding values in the others. As the present complex is the only red one among the otherwise yellow complexes of Table 5, one is tempted to conclude that the "abnormal" colour of the former is associated with the large deviation from 90° of the bond angles at tellurium. A simple explanation of the tellurium-ligand bonds in square-planar complexes of divalent tellurium is provided by the three-centre, two-electron-

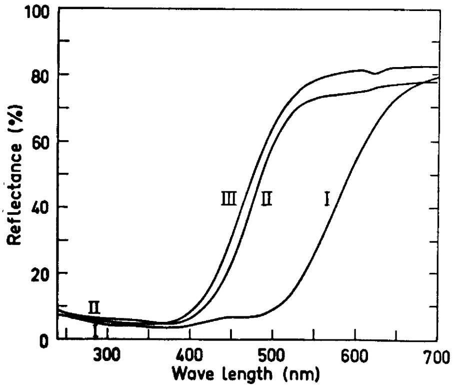

Fig. 1. Visible and ultraviolet reflectance spectra of solid $Te(tmtu)_2(S_2O_2C_6H_5)_2$ (I), $Te(trtu)_2$-$(S_2O_2C_6H_5)_2$ (II), and $Te(etu)_2(S_2O_2C_6H_5)_2$ (III), with MgO as a white standard of 100 % reflectance.

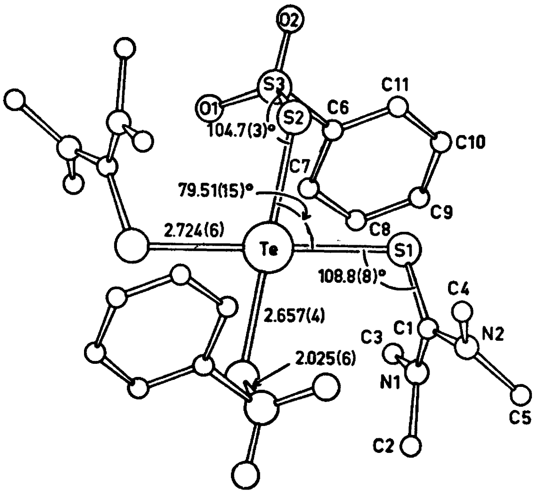

Fig. 2. *trans*-Dibenzenethiosulphonatobis(tetramethylthiourea)tellurium(II) as seen normal to the plane of the TeS_4 coordination group.

pair bonding scheme, based on tellurium *5p* orbitals. This would imply a S – Te – S angle of 90°. The observed value, 79.51(15)°, which probably is the result of the steric requirements of the rather bulky ligands, does not fit as well into this picture as is normally the case. Nevertheless, the tellurium-sulphur bond lengths indicate that these bonds are essentially of the same kind as in other square-planar complexes of divalent tellurium.

The benzenethiosulphonate S – S bond length is in good agreement with the corresponding bond lengths in the other complexes of Table 5. Bond lengths and angles between light atoms are in the normal range. With the sulphur coordinates given three times the weight of the carbon and nitrogen coordinates, the atoms of a least squares plane through S(3) and the benzene ring deviate 0.000 – 0.017 Å from the plane. The atoms of a least squares plane through the thiourea part of the tetramethylthiourea group deviate 0.004 – 0.047 Å from the plane. The angle between the latter plane and a plane through the TeS_4 coordination group is 61.0°. The methyl carbon atoms, C(2), C(3), C(4), and C(5) are – 0.554, 0.364, – 0.492, and 0.742 Å, respectively, distant from the plane through the thiourea part of the tetramethylthiourea group. This plane makes an angle of 22.1° with a least squares plane through N(1) and the carbon atoms bonded to N(1), and an angle of 30.1° with a least squares plane through N(2) and the carbon atoms bonded to N(2).

The steric requirements of the ligands lead to short non-bonding, intramolecular distances. (A prime denotes an atom generated by the centre of symmetry). The distances S(2) – C(2′) = 3.747 Å, S(2) – C(3′) = 3.779 Å, O(1) – C(1′) = 3.189 Å, and O(1) – C(4′) = 3.145 Å imply that the S(1′) – Te – S(2) angle, which is 100.49°, can hardly be decreased without a reorientation of the ligands. As will be seen from Fig. 2, the

Table 5. Principal bond lengths (Å) and angles (°) in complexes of tellurium dibenzenethiosulphonate with thioureas. Standard deviations in parentheses.

Complex	Reference	Te – S (thio-sulphonate)	Te – S (thio-ureas)	∠S – Te – S	S – S
$Te(tmtu)_2(S_2O_2C_6H_5)_2$	Present	2.657(4)	2.724(6)	79.51(15)	2.025(6)
$Te(trtu)_2(S_2O_2C_6H_5)_2$	3	2.668(3)	2.691(4)	88.85(9)	2.018(4)
$Te(etu)_2(S_2O_2C_6H_5)_2$	4	2.686(4)	2.713(5)	90.80(13)	2.016(5)

area around the tellurium atom is rather crowded. In addition to the distances just mentioned, some other, short distances within the molecule are: Te – C(3) = 3.968 Å, Te – C(6) = 3.971 Å, Te – C(7) = 3.894 Å, S(2) – C(11) = 3.659 Å, O(1) – C(7) = 2.888 Å, O(2) = C(11) – 2.992 Å, C(3) – C(8) = 3.592 Å. No intermolecular distances are essentially shorter than the sum of the appropriate van der Waals radii.[10] It may therefore be concluded that the deviation of the S – Te – S angle from 90° is caused mainly by strain within the molecule, and not by crystal packing effects.

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