

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

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The complex *trans*-dibenzenethiosulphonato-bis(trimethylenethiourea)tellurium(II), $\text{Te}(\text{S}:\text{C}\cdot\text{NH}\cdot[\text{CH}_2]_3\cdot\text{NH})_2(\text{S}_2\text{O}_2\text{C}_6\text{H}_5)_2$, crystallizes in the space group $P\bar{1}$ (No. 2) with unit cell dimensions: $a = 8.790 \text{ \AA}$, $b = 10.178 \text{ \AA}$, $c = 8.004 \text{ \AA}$, $\alpha = 97.40^\circ$, $\beta = 96.16^\circ$, $\gamma = 99.96^\circ$. The measured density is 1.69 g/cm^3 , and the calculated density, with one molecule per unit cell, is 1.69 g/cm^3 . The crystal and molecular structure has been determined by three-dimensional X-ray methods. The intensity data of 1692 independent, non-zero reflections were collected using integrated Weissenberg techniques. Least squares refinement procedures resulted in a conventional R -value of 0.061.

The tellurium atoms lie in centres of symmetry, and are bonded to two trimethylenethiourea sulphur atoms and two benzenethiosulphonate sulphur atoms in a *trans* square-planar arrangement. The TeS_4 group has the dimensions, with standard deviations in parentheses: $\text{Te}-\text{S}(\text{trimethylenethiourea}) = 2.691(4) \text{ \AA}$, $\text{Te}-\text{S}(\text{benzenethiosulphonate}) = 2.668(3) \text{ \AA}$, $\angle \text{S}-\text{Te}-\text{S} = 88.85(9)^\circ$. The benzenethiosulphonate S—S bond is $2.018(4) \text{ \AA}$. These dimensions may be explained in terms of a three-centre two-electron-pair bonding scheme, based on tellurium $5p$ orbitals.

This work forms part of a series of structure studies on square-planar complexes of divalent tellurium. Among the complexes of the type TeL_2X_2 , the only thiosulphonate complex whose structure has been reported earlier is *trans*-dimethanethiosulphonato-bis(thiourea)tellurium(II), $\text{Te}(\text{tu})_2(\text{S}_2\text{O}_2\text{CH}_3)_2$.¹ The present work on *trans*-dibenzenethiosulphonato-bis(trimethylenethiourea)tellurium(II), $\text{Te}(\text{S}:\text{C}\cdot\text{NH}\cdot[\text{CH}_2]_3\cdot\text{NH})_2(\text{S}_2\text{O}_2\text{C}_6\text{H}_5)_2$, was undertaken to get more data on the S—S bond in the thiosulphonate part of the complex. This bond length can be understood on the basis of the bonding around tellurium. The S—S bond in the complex may be compared with S—S bond lengths in uncomplexed tellurium thiosulphonates, and in ionic sodium methanethiosulphonate.

EXPERIMENTAL

The benzenethiosulphonato complex, $\text{Te}(\text{trtu})_2(\text{S}_2\text{O}_3\text{C}_6\text{H}_5)_2$, where trtu = trimethylenethiourea, was prepared from the chloro complex, as follows.

0.86 g dichloro-bis(trimethylenethiourea)tellurium(II) ² and 1.24 g potassium benzenethiosulphonate dihydrate (25 % excess) were dissolved, by stirring and heating on a waterbath, in 3 ml of dimethylformamide. The resulting red solution was cooled to room temperature, 10 ml of methanol were added, and the now a little opaque solution was filtered by suction through a glass filter, while still at room temperature. Crystallization from the red filtrate set in on seeding or on scratching of the beaker walls. Yield, after cooling to about 0° in a refrigerator, about 1.1 g (78 %). The crystals were drained well on the filter, and washed with dry ether. They occur as well developed, yellow, triclinic prisms bounded by {110}, {1 $\bar{1}$ 0}, {010}, and {01 $\bar{1}$ }. M.p. 146–148° (decomp.). (Found: Te 18.06. Calc. for $\text{C}_{30}\text{H}_{24}\text{N}_4\text{O}_4\text{S}_6\text{Te}$: Te 18.06.)

Unit cell dimensions were determined from zero-layer Weissenberg photographs taken at room temperature around the a , b , and c axes, using $\text{CuK}\alpha_1$ radiation ($\lambda = 1.5405$ Å). Sodium chloride ($a = 5.6394$ Å) was used as an internal standard.³ 2θ -values were measured for 86 reflections.

Intensity data were collected for the $0kl-5kl$, $h0l$ and $hk0$ layers, using multifold integrating equi-inclination Weissenberg techniques with (Ni-filtered) $\text{CuK}\alpha$ -radiation. Crystal dimensions are shown in Table 1. Intensity measurements were made visually

Table 1. Distances (mm) from origin to faces for the three crystals used for collecting intensity data.

Distance to	Crystal rotating about the a axis	Crystal rotating about the b axis	Crystal rotating about the c axis
{110} and {1 $\bar{1}$ 0}	0.0594	0.0555	0.0488
{1 $\bar{1}$ 0} and {110}	0.0962	0.0763	0.0644
{010} and {0 $\bar{1}$ 0}	0.0367	0.0456	0.0432
{01 $\bar{1}$ } and {0 $\bar{1}$ 1}	0.0388	0.0636	0.0384

with a scale of timed exposures having the same spot shape. Out of 1993 accessible, independent reflections, 1692 were strong enough to be measured. The intensities of the remaining 301 reflections were set equal to the observable limit. Estimated corrections for the splitting of α_1 and α_2 at high angles were applied.

Absorption and Lorentz-polarization corrections were done ($\mu = 132$ cm⁻¹). The absorption correction method described by Busing and Levy,⁴ and modified by Coppens, Leiserowitz and Rabinovich,⁵ was applied, using a $6 \times 6 \times 6$ grid for each of the crystals.

The calculated structure factors were based on the scattering curves listed in *International Tables* (Ref. 3, p. 202). The tellurium and sulphur scattering curves were corrected for anomalous dispersion, using the Af' and Af'' values given by Cromer.⁶

The least squares refinement was carried out with a full-matrix program minimizing the function

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

where K is the scale factor and $W = 1/[(K\alpha_1)^2 + (\alpha_2 F_o)^2/4W_o]$. Here W_o is an individual weight, related to the reliability with which the intensities were measured, and α_1 and α_2 are constants. Non-observed reflections for which $|F_c|$ 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 use on the IBM computer by Dr. Dove Rabinovich. Two of the programs used, one of them correcting for extinction, and the other one for calculating weighted least squares planes, were written by Mr. Knut Maartmann-Moe, of this Institute.

CRYSTAL DATA

The crystals of *trans*-dibenzenethiosulphonato-bis(trimethylenethiourea)-tellurium(II), $\text{Te}(\text{trtu})_2(\text{S}_2\text{O}_2\text{C}_6\text{H}_5)_2$, are triclinic. Based on 86 measured 2θ -values a least squares procedure gave the following unit cell dimensions.

$$\begin{array}{lll} a = 8.790(2) \text{ \AA}; & b = 10.178(2) \text{ \AA}; & c = 8.004(2) \text{ \AA}; \\ \alpha = 97.40(4)^\circ; & \beta = 96.16(5)^\circ; & \gamma = 99.96(3)^\circ. \end{array}$$

The uncertainties given in parentheses are standard deviations based on least squares, neglecting uncertainties in film radii.

$$V = 693.1 \text{ \AA}^3; \quad M = 706.44; \quad F(000) = 328; \quad Z = 1.$$

The density obtained by flotation is 1.69 g/cm^3 , the calculated density is 1.69 g/cm^3 .

Possible space groups: $P1$ (No. 1) and $P\bar{1}$ (No. 2).

STRUCTURE DETERMINATION

Assuming space group $P\bar{1}$ (No. 2), which requires that the tellurium atom lies in a centre of symmetry, the structure was solved in a straight-forward way through two-dimensional Fourier synthesis based on strong reflections with positive signs, working simultaneously with projections along the a , b , and c axis.

After a second Fourier synthesis, least squares refinement was started for the $0kl$, $h0l$ and $hk0$ reflections. Scale factors, positional parameters and isotropic thermal parameters for all non-hydrogen atoms were refined, to an R value of about 0.10. All reflections were then included in the refinement. After a few cycles of refinement on the parameters mentioned above, anisotropic temperature factors were introduced for tellurium and sulphur atoms, and scale factors were excluded from the refinement. The R value dropped to 0.069.

The data were then corrected for extinction according to Zachariasen.⁷ The form

$$F_{\text{corr}} = KF_{\text{obs}}[1 + \beta(2\theta)CI_{\text{obs}}]$$

was used, where F_{obs} is the observed structure factor, F_{corr} the value corrected for secondary extinction, I_{obs} the observed integrated intensity, K and C scale factors to be adjusted and $\beta(2\theta) = 2(1 + \cos^4 2\theta)/(1 + \cos^2 2\theta)^2$. The absorption term in $\beta(2\theta)$ was neglected. With I_{obs} on an absolute scale, the value of C was found to be 3.7×10^{-6} .

After some additional cycles, where an overall scale factor was introduced in the refinement, no shifts were greater than 0.16 times the standard deviation, and the refinement was terminated. The constants a_1 and a_2 in the weighting scheme were both put equal to unity during the last refinement cycles. The final value of R , including non-observed reflections when $|F_c|$ exceeds the observable limit, is 0.061. A final difference Fourier summation for the projection along the a axis showed no peaks higher than 1.0 e/\AA^2 outside

the expected hydrogen positions. The successful refinement indicates that the choice of space group $P\bar{1}$ (No. 2) is the correct one.

The final atomic coordinates are listed in Table 2, together with the final isotropic thermal parameters for the light atoms. The final anisotropic thermal parameters for the tellurium and sulphur atoms are listed in Table 3. The structure factors calculated from the final parameters are listed in Table 4.

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

	x	y	z	U
Te	0	0	0	
S(1)	-0.25482(34)	0.05375(26)	0.13331(35)	
S(2)	0.14950(34)	0.24788(24)	0.13650(32)	
S(3)	0.26808(34)	0.22742(26)	0.35891(32)	
O(1)	0.4281(10)	0.2918(8)	0.3604(11)	0.0688(24)
O(2)	0.2413(10)	0.0891(8)	0.3896(10)	0.0614(21)
N(1)	-0.4288(11)	0.2268(9)	0.0518(12)	0.0499(23)
N(2)	-0.3220(10)	0.1264(8)	-0.1708(11)	0.0443(20)
C(1)	-0.3382(12)	0.1419(9)	-0.0086(13)	0.0402(23)
C(2)	-0.3987(15)	0.1972(12)	-0.2967(16)	0.0630(33)
C(3)	-0.4195(14)	0.3342(11)	-0.2076(15)	0.0566(30)
C(4)	-0.5088(15)	0.3065(12)	-0.0569(16)	0.0613(32)
C(5)	0.1883(12)	0.3208(10)	0.5195(13)	0.0421(24)
C(6)	0.0894(13)	0.2532(11)	0.6134(14)	0.0500(27)
C(7)	0.0262(16)	0.3304(13)	0.7383(17)	0.0676(35)
C(8)	0.0654(16)	0.4701(13)	0.7643(16)	0.0648(33)
C(9)	0.1665(14)	0.5353(11)	0.6659(15)	0.0542(29)
C(10)	0.2311(13)	0.4618(11)	0.5407(14)	0.0496(26)

Table 3. Anisotropic thermal parameters (\AA^2) in the form $\exp[-2\pi^2(h^2a^{-2}U_{11} + \dots + 2hka^{-1}b^{-1}U_{12} + \dots)]$.

All values have been multiplied by 10^4 . Standard deviations are given in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Te	346(6)	356(5)	340(5)	101(4)	49(4)	20(5)
S(1)	525(20)	556(15)	498(17)	155(14)	148(13)	127(16)
S(2)	568(20)	444(13)	393(15)	96(13)	13(11)	-5(15)
S(3)	498(19)	555(15)	377(14)	146(14)	-40(12)	18(14)

RESULTS

Bond lengths and angles, as calculated from the coordinates of Table 2, are listed in Table 5. Coordinate covariances and standard deviations in unit cell dimensions have been neglected. When calculating weighted least squares planes through groups of atoms, the sulphur atoms were given four times the weight of the lighter atoms. A drawing of the molecule, with the principal bond lengths and angles, is reproduced in Fig. 1.

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

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
0	1	0	651	622	0	0	9	105	113	0	4	-9	138	135	1	2	8	-103	50
0	2	0	240	-207	0	1	9	53	-21	0	5	-9	221	215	1	3	8	248	257
0	3	0	370	374	0	2	9	80	85	0	6	-9	165	174	1	4	8	179	199
0	4	0	782	823	0	3	9	157	156	0	7	-9	43	48	1	5	8	-73	73
0	5	0	140	125	0	4	9	97	99	0	1	-10	109	113	1	10	9	-89	74
0	6	0	649	618	0	5	-1	949	1089	0	2	-10	110	124	1	1	9	86	106
0	7	0	78	81	0	2	-1	739	716	0	3	-10	85	101	1	2	9	136	194
0	8	0	242	229	0	3	-1	330	291	0	4	-10	48	51	1	3	9	108	146
0	9	0	243	255	0	4	-1	407	412	1	0	-1	158	-187	1	0	-1	496	567
0	10	0	-50	7	0	5	-1	840	824	1	2	0	935	940	1	1	-1	992	1085
0	11	0	-47	15	0	6	-1	400	422	1	3	0	915	913	1	2	-1	265	231
0	12	0	99	98	0	7	-1	142	137	1	4	0	584	583	1	3	-1	518	461
0	0	1	550	538	0	8	-1	192	238	1	5	0	98	93	1	4	-1	529	540
0	1	1	639	577	0	9	-1	150	145	1	6	0	242	225	1	5	-1	444	442
0	2	1	209	185	0	10	-1	43	44	1	7	0	398	389	1	6	-1	161	172
0	3	1	413	622	0	11	-1	-47	22	1	8	0	170	390	1	7	-1	244	247
0	4	1	277	241	0	12	-1	41	54	1	9	0	111	126	1	8	-1	306	303
0	5	1	89	-87	0	1	-2	450	467	1	10	0	48	37	1	9	-1	293	246
0	6	1	40	72	0	2	-2	514	446	1	11	0	163	154	1	10	-1	199	192
0	7	1	365	374	0	3	-2	305	328	1	12	0	112	128	1	11	-1	115	104
0	8	1	277	248	0	4	-2	367	391	1	0	1	105	-48	1	12	-1	100	92
0	9	1	-48	41	0	5	-2	511	555	1	1	1	739	760	1	0	-2	63	18
0	10	1	-51	55	0	6	-2	461	458	1	2	1	1118	1121	1	1	-2	443	438
0	11	1	117	114	0	7	-2	296	297	1	3	1	682	678	1	2	-2	301	278
0	12	1	12	135	0	8	-2	246	277	1	4	1	513	513	1	3	-2	77	-41
0	0	2	303	321	0	9	-2	237	250	1	5	1	364	364	1	4	-2	304	336
0	1	2	515	527	0	10	-2	104	108	1	6	1	337	320	1	5	-2	338	339
0	2	2	304	336	0	11	-2	-47	12	1	7	1	314	319	1	6	-2	329	312
0	3	2	414	423	0	12	-2	73	88	1	8	1	-90	43	1	7	-2	265	252
0	4	2	254	225	0	1	-3	680	709	1	9	1	72	64	1	8	-2	343	369
0	5	2	41	42	0	2	-3	239	266	1	10	1	144	135	1	9	-2	271	253
0	6	2	124	103	0	3	-3	136	122	1	11	1	157	158	1	10	-2	164	147
0	7	2	261	269	0	4	-3	420	461	1	12	1	-50	19	1	11	-2	129	127
0	8	2	68	82	0	5	-3	613	576	1	0	2	530	517	1	12	-2	127	117
0	9	2	71	85	0	6	-3	446	420	1	1	2	535	503	1	0	-3	145	106
0	10	2	175	174	0	7	-3	183	202	1	2	2	598	610	1	1	-3	235	229
0	11	2	167	171	0	8	-3	133	127	1	3	2	545	535	1	2	-3	151	142
0	12	2	63	73	0	9	-3	250	252	1	4	2	105	94	1	3	-3	128	225
0	0	3	424	405	0	10	-3	268	249	1	5	2	295	315	1	4	-3	203	61
0	1	3	107	-87	0	11	-3	88	96	1	6	2	245	271	1	5	-3	188	177
0	2	3	55	-24	0	12	-3	34	44	1	7	2	-89	43	1	6	-3	-83	14
0	3	3	201	201	0	1	-4	134	148	1	8	2	-93	40	1	7	-3	220	230
0	4	3	240	283	0	2	-4	422	444	1	9	2	98	105	1	8	-3	248	251
0	5	3	177	174	0	3	-4	279	262	1	10	2	174	146	1	9	-3	245	258
0	6	3	123	120	0	4	-4	123	104	1	11	2	104	72	1	10	-3	140	119
0	7	3	213	212	0	5	-4	242	262	1	0	3	110	114	1	11	-3	137	116
0	8	3	229	230	0	6	-4	511	503	1	1	3	336	313	1	12	-3	115	116
0	9	3	178	158	0	7	-4	311	325	1	2	3	557	556	1	1	-4	473	467
0	10	3	155	152	0	8	-4	47	43	1	3	3	255	252	1	2	-4	462	449
0	11	3	123	114	0	9	-4	227	231	1	4	3	334	-34	1	3	-4	368	39
0	0	4	148	-114	0	10	-4	273	244	1	5	3	163	169	1	4	-4	105	101
0	1	4	97	-44	0	11	-4	143	160	1	6	3	228	244	1	5	-4	299	278
0	2	4	517	521	0	12	-4	58	62	1	7	3	196	183	1	6	-4	249	285
0	3	4	238	280	0	1	-5	75	-62	1	8	3	-98	68	1	7	-4	50	5
0	4	4	75	41	0	2	-5	265	270	1	9	3	110	105	1	8	-4	90	8
0	5	4	185	159	0	3	-5	262	277	1	10	3	93	85	1	9	-4	188	150
0	6	4	468	497	0	4	-5	66	51	1	11	3	93	87	1	10	-4	184	175
0	7	4	242	260	0	5	-5	91	-82	1	0	4	282	254	1	11	-4	86	85
0	8	4	139	127	0	6	-5	134	165	1	1	4	589	593	1	1	-5	325	312
0	9	4	90	91	0	7	-5	164	166	1	2	4	434	453	1	2	-5	440	512
0	10	4	133	133	0	8	-5	270	269	1	3	4	-73	-48	1	3	-5	270	275
0	0	5	294	281	0	9	-5	118	114	1	4	4	-80	15	1	4	-5	181	168
0	1	5	644	630	0	10	-5	120	132	1	5	4	218	205	1	5	-5	-84	55
0	2	5	181	196	0	11	-5	139	142	1	6	4	277	266	1	6	-5	155	145
0	3	5	75	70	0	1	-6	292	282	1	7	4	-98	-29	1	7	-5	187	169
0	4	5	175	190	0	2	-6	117	130	1	8	4	-101	40	1	8	-5	-95	88
0	5	5	371	379	0	3	-6	112	109	1	9	4	169	174	1	9	-5	-95	-19
0	6	5	184	209	0	4	-6	104	114	1	10	4	182	177	1	10	-5	149	136
0	7	5	179	162	0	5	-6	128	119	1	0	5	411	419	1	11	-5	129	123
0	8	5	92	97	0	6	-6	114	113	1	1	5	245	246	1	2	-6	335	345
0	9	5	184	170	0	7	-6	151	160	1	2	5	-77	-57	1	3	-6	579	550
0	10	5	94	113	0	8	-6	117	82	1	3	5	-82	42	1	4	-6	310	292
0	0	6	408	392	0	9	-6	64	61	1	4	5	268	288	1	5	-6	-91	22
0	1	6	233	259	0	10	-6	80	85	1	5	5	199	209	1	6	-6	84	72
0	2	6	131	133	0	11	-6	97	108	1	6	5	-108	44	1	7	-6	204	172
0	3	6	189	186	0	1	-7	203	219	1	7	5	-102	55	1	8	-6	-98	69
0	4	6	310	323	0	2	-7	182	201	1	8	5	174	170	1	9	-6	-90	80
0	5	6	212	213	0	3	-7	135	133	1	9	5	216	216	1	10	-6	74	65
0	6	6	105	98	0	4	-7	102	95	1	0	6	-83	40	1	11	-6	61	65
0	7	6	95	95	0	5	-7	131	144	1	1	6	170	150	1	4	-7	244	239
0	8	6	191	193	0	6	-7	108	99	1	2	6	169	186	1	5	-7	170	171
0	9	6	138	146	0	7	-7	132	127	1	3	6	199	214	1	6	-7	195	155
0	0	7	192	194	0	8	-7	121	113	1	4	6	188	186	1	7	-7	221	196
0	1	7	321	322	0	9	-7	-41	-35	1	5	6	214	199	1	8	-7	144	132
0	2	7	290	280	0	10	-7	-31	12	1	6	6	-103	74	1	9	-7	176	65
0	3	7	204	194	0	1	-8	181	194	1	7	6	145	132	1	0	-8	462	393
0	4	7	172	157	0	2	-8												

DIVALENT TELLURIUM COMPLEX

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)						
1	-2	10	96	110	2	5	3	313	322	2	5	-5	466	461	2	-2	7	157	163	3	5	1	154	134	
1	-3	10	91	114	2	6	3	271	256	2	6	-5	384	404	2	-3	7	-88	59	3	6	1	172	143	
1	-1	-1	118	85	2	7	3	130	143	2	7	-5	143	141	2	-4	7	162	158	3	7	1	139	133	
1	-2	-1	514	-523	2	8	3	-91	46	2	8	-5	-87	30	2	-5	-7	310	300	3	8	1	198	201	
1	-3	-1	45	77	2	9	3	96	90	2	9	-5	142	112	2	-6	7	186	159	3	9	1	233	223	
1	-4	-1	380	394	2	10	3	106	110	2	10	-5	110	112	2	-7	7	91	80	3	10	1	129	125	
1	-5	-1	360	347	2	0	4	632	641	2	11	-5	-54	42	2	-8	7	152	141	3	11	1	61	68	
1	-6	-1	-76	31	2	1	4	489	503	2	2	-6	360	367	2	-9	7	229	232	3	0	2	139	-135	
1	-7	-1	184	195	2	2	4	252	242	2	3	-6	265	259	2	-10	7	127	129	3	1	2	361	374	
1	-8	-1	389	415	2	3	4	104	95	2	4	-6	155	135	2	-1	8	141	150	3	2	2	421	451	
1	-9	-1	248	246	2	4	4	312	332	2	5	-6	274	282	2	-2	8	200	210	3	3	2	97	66	
1	-10	-1	98	97	2	5	4	357	343	2	6	-6	314	301	2	-3	8	-94	32	3	4	2	111	103	
1	-11	-1	129	124	2	6	4	-87	36	2	7	-6	220	210	2	-4	8	-21	-21	3	5	2	339	356	
1	-12	-1	181	177	2	7	7	91	-64	2	8	-6	125	106	2	-5	8	157	143	3	6	2	295	281	
1	-1	-2	212	224	2	8	4	135	118	2	9	-6	88	84	2	-6	8	182	180	3	7	2	189	195	
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1	-3	-2	671	661	2	0	5	323	297	2	4	-7	268	245	2	-8	8	101	100	3	9	2	183	163	
1	-4	-2	155	162	2	1	5	87	67	2	5	-7	199	202	2	-9	8	110	130	3	10	2	154	148	
1	-5	-2	112	112	2	2	5	112	112	2	6	-7	162	141	2	-1	9	-78	28	3	0	3	459	467	
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1	-8	-2	232	253	2	5	5	-89	22	2	9	-7	120	118	2	-4	9	-74	36	3	3	3	148	137	
1	-9	-2	96	100	2	6	5	-94	53	2	10	-7	588	606	2	-5	9	-49	-45	3	4	3	283	264	
1	-10	-2	184	187	2	7	6	137	134	2	11	-7	30	325	2	-6	9	67	72	3	5	3	455	476	
1	-11	-2	218	211	2	8	5	158	147	2	12	-7	0	438	439	2	-7	9	99	114	3	6	3	227	222
1	-12	-2	121	140	2	9	5	-53	20	2	13	-7	0	240	247	2	-1	-1	1015	1053	3	7	3	-97	-10
1	-1	-3	486	484	2	0	6	175	161	2	4	-8	363	350	2	-2	-1	369	415	3	8	3	199	169	
1	-2	-3	672	680	2	1	6	187	190	2	5	-8	514	529	2	-3	-1	271	528	3	9	3	191	204	
1	-3	-3	320	320	2	2	7	-82	71	2	6	-8	311	304	2	-4	-1	583	542	3	10	3	85	85	
1	-4	-3	330	319	2	3	6	162	140	2	7	-8	0	178	162	2	-5	-1	676	732	3	0	4	473	483
1	-5	-3	305	318	2	4	6	92	106	2	8	-8	0	262	277	2	-6	-1	250	290	3	1	4	263	207
1	-6	-3	390	385	2	5	6	151	130	2	9	-8	0	286	287	2	-7	-1	95	107	3	2	4	83	76
1	-7	-3	158	167	2	6	6	-60	65	2	10	-8	0	117	121	2	-8	-3	159	171	3	3	4	275	286
1	-8	-3	409	211	2	7	6	151	131	2	11	-8	0	92	104	2	-9	-1	376	373	3	4	4	395	396
1	-9	-3	112	129	2	8	6	-52	58	2	12	-8	1	157	-66	2	-10	-1	229	205	3	5	4	201	207
1	-10	-3	188	173	2	0	7	187	169	2	1	-9	1	367	358	2	-11	-1	-85	54	3	6	4	-97	70
1	-11	-3	131	131	2	1	7	-91	-30	2	2	-9	1	126	108	2	-12	-1	71	72	3	7	4	220	184
1	-12	-3	218	217	2	2	7	-3	13	2	3	-9	1	148	173	2	-1	-2	352	250	3	8	5	209	209
1	-1	-4	278	337	2	3	7	135	122	2	4	-9	1	94	84	2	-2	-2	271	-210	3	9	4	104	105
1	-3	-4	644	635	2	4	7	149	139	2	5	-9	1	285	251	2	-3	-2	397	462	3	0	5	375	365
1	-4	-4	412	408	2	5	7	-84	61	2	6	-9	1	75	82	2	-4	-2	975	1010	3	1	5	208	207
1	-5	-4	168	154	2	6	7	97	89	2	7	-9	1	214	239	2	-5	-2	459	456	3	2	5	510	277
1	-6	-4	218	218	2	7	7	-94	54	2	8	-9	1	381	381	2	-6	-3	158	171	3	3	5	314	304
1	-7	-4	400	294	2	8	7	-93	35	2	9	-9	1	278	254	2	-7	-2	322	319	3	4	5	204	205
1	-8	-4	-101	104	2	9	7	132	177	2	10	-9	1	155	152	2	-8	-2	446	439	3	5	5	-100	67
1	-9	-4	-99	92	2	10	7	163	152	2	11	-9	1	85	91	2	-9	-2	179	162	3	6	5	212	195
1	-10	-4	108	116	2	11	7	-71	49	2	12	-9	1	723	742	2	-10	-2	-91	25	3	7	5	147	158
1	-11	-4	86	86	2	12	7	-85	85	2	13	-9	1	820	851	2	-11	-2	80	80	3	8	6	79	79
1	-1	-5	406	456	2	0	8	-75	47	2	14	-9	1	207	144	2	-12	-2	102	100	3	0	6	403	399
1	-2	-5	602	654	2	1	8	155	158	2	15	-9	1	303	-264	2	-1	-3	275	290	3	1	6	281	247
1	-3	-5	337	329	2	2	8	155	163	2	16	-9	1	289	310	2	-2	-3	681	688	3	2	6	114	112
1	-4	-5	-84	82	2	3	8	-299	303	2	17	-9	1	314	325	2	-3	-3	585	559	3	3	6	147	129
1	-5	-5	-17	20	2	4	8	-374	40	2	18	-9	1	88	83	2	-4	-3	358	347	3	4	6	191	164
1	-6	-5	324	321	2	5	8	-59	21	2	19	-9	1	-81	44	2	-5	-3	202	192	3	5	6	99	104
1	-7	-5	162	147	2	6	8	422	433	2	20	-9	1	167	161	2	-6	-3	352	367	3	6	6	-86	29
1	-8	-5	-101	-36	2	7	8	-445	418	2	21	-9	1	298	292	2	-7	-3	168	177	3	7	6	74	89
1	-9	-5	-86	17	2	8	8	-73	59	2	22	-9	1	159	144	2	-8	-3	198	172	3	8	7	-97	7
1	-10	-5	96	103	2	9	8	-72	6	2	23	-9	1	-68	44	2	-9	-3	-83	3	9	7	-101	56	
1	-1	-6	256	252	2	10	8	217	202	2	24	-9	1	138	118	2	-10	-3	-88	59	3	2	7	205	194
1	-3	-6	-87	-30	2	11	8	285	308	2	25	-9	1	377	395	2	-11	-3	98	99	3	3	7	219	210
1	-4	-6	112	116	2	12	8	136	136	2	26	-9	1	550	599	2	-12	-4	562	552	3	4	7	-92	5
1	-5	-6	288	297	2	13	8	101	2	2	27	-9	1	-42	32	2	-1	-4	212	210	3	5	7	-78	7
1	-6	-6	218	215	2	14	8	150	151	2	28	-9	1	108	-120	2	-3	-4	461	369	3	6	7	62	79
1	-7	-6	-101	-40	2	15	8	132	162	2	29	-9	1	258	254	2	-4	-4	239	243	3	0	8	-97	72
2	0	0	77	-97	2	16	8	771	772	2	30	-9	1	394	412	2	-5	-4	137	114	3	1	8	155	132
2	1	0	635	-600	2	17	8	587	575	2	31	-9	1	130	115	2	-6	-4	-84	24	3	2	8	191	176
2	2	0	71	30	2	18	8	-45	44	2	32	-9	1	-86	4	2	-7	-4	176	166	3	3	2	-77	55
2	3	0	463	695	2	19	8	182	182	2	33	-9	1	107	103	2	-8	-4	93	97	3	4	8	-59	3
2	4	0	279	283	2	20	8																		

Table 4. Continued.

H	K	L	F(0)	F(1)	H	K	L	F(0)	F(1)	H	K	L	F(0)	F(1)	H	K	L	F(0)	F(1)	H	K	L	F(0)	F(1)
3	0	-4	286	296	3	10	-5	-86	41	4	7	0	48	32	4	2	-4	136	126	4	-7	6	-93	37
3	0	-4	466	411	3	-11	5	76	73	4	8	0	93	105	4	3	-4	189	164	4	-8	6	-89	-10
3	2	-4	298	287	3	-1	6	307	408	4	0	0	115	119	4	4	-4	161	175	4	-9	6	132	128
3	3	-4	228	253	3	-2	6	194	189	4	10	0	109	106	4	5	-4	173	180	4	-10	3	153	126
3	4	-4	423	419	3	-3	6	-83	37	4	0	1	304	322	4	6	-4	162	181	4	-1	7	106	86
3	5	-4	278	250	3	-4	6	292	291	4	1	1	272	252	4	7	-4	177	177	4	-2	7	281	263
3	6	-4	176	169	3	-5	6	322	302	4	2	1	324	291	4	8	-4	201	186	4	-3	7	210	220
3	7	-4	235	235	3	-6	6	-95	78	4	3	1	434	396	4	9	-4	181	146	4	-4	7	-94	70
3	8	-4	335	295	3	-7	6	-68	2	4	4	1	196	158	4	10	-4	78	354	4	-5	7	-64	74
3	9	-4	112	104	3	-8	6	170	157	4	5	1	148	129	4	1	-5	89	94	4	-6	7	176	156
3	10	-4	-79	76	3	-9	6	140	137	4	6	1	147	140	4	2	-5	85	67	4	-7	7	140	129
3	11	-4	63	82	3	-10	6	-73	53	4	7	1	-86	84	4	3	-5	183	189	4	-8	7	-73	50
3	1	-5	323	307	3	-1	7	352	316	4	8	1	-90	77	4	4	-5	187	178	4	-9	7	-58	-21
3	2	-5	149	159	3	-2	7	254	239	4	9	1	92	100	4	5	-5	85	74	4	-1	8	84	7
3	3	-5	114	115	3	-3	7	154	127	4	10	1	77	77	4	6	-5	-88	-16	4	-2	8	160	137
3	4	-5	298	302	3	-4	7	148	124	4	0	2	366	388	4	7	-5	-89	61	4	-3	8	229	217
3	5	-5	298	291	3	-5	7	269	233	4	1	2	448	488	4	8	-5	195	174	4	-4	8	160	150
3	6	-5	182	162	3	-6	7	356	307	4	2	2	259	228	4	9	-5	127	120	4	-5	8	111	117
3	7	-5	175	152	3	-7	7	186	146	4	3	2	66	-17	4	10	-5	69	72	4	-6	8	-71	60
3	8	-5	205	195	3	-8	7	-85	17	4	4	2	258	235	4	2	-6	93	110	4	-7	8	60	66
3	9	-5	231	219	3	-9	7	79	84	4	5	2	260	249	4	3	-6	-86	-6	4	-8	8	42	54
3	10	-5	105	101	3	-10	7	114	136	4	6	2	103	117	4	4	-6	171	173	4	-1	9	82	93
3	11	-5	42	45	3	-1	8	-98	48	4	7	2	-90	30	4	5	-6	213	237	4	-2	9	100	118
3	2	-6	191	174	3	-2	8	191	169	4	8	1	89	42	4	6	-6	-92	-1	4	-3	9	130	120
3	3	-6	-88	24	3	-3	8	226	202	4	9	2	151	142	4	7	-6	-88	-40	4	-4	9	118	135
3	4	-6	-91	-79	3	-4	8	107	98	4	10	2	80	90	4	8	-6	-78	69	4	-1	-1	290	305
3	5	-6	175	182	3	-5	8	138	118	4	0	3	285	272	4	9	-6	123	138	4	-2	-1	230	235
3	6	-6	299	276	3	-6	8	203	192	4	1	3	133	117	4	5	-7	-94	54	4	-3	-1	275	271
3	7	-6	330	316	3	-7	8	191	180	4	2	3	133	-109	4	6	-7	160	141	4	-4	-1	235	206
3	8	-6	111	102	3	-8	8	91	89	4	3	3	237	219	4	-1	0	390	477	4	-5	-1	176	161
3	9	-6	133	126	3	-1	9	-73	58	4	4	3	300	280	4	2	0	465	480	4	-6	-1	169	165
3	10	-6	140	148	3	-2	9	-75	53	4	5	3	103	81	4	-3	0	378	382	4	-7	-1	287	295
3	5	-7	-100	-10	3	-3	9	117	115	4	6	3	-90	-39	4	-4	0	117	124	4	-8	-1	86	87
3	6	-7	184	119	3	-4	9	114	114	4	7	3	166	157	4	-5	-5	311	321	4	-9	-1	-91	62
3	7	-7	159	152	3	-5	9	115	114	4	8	3	250	241	4	-6	0	334	350	4	-10	-1	169	166
3	8	-7	-78	72	3	-6	9	109	125	4	9	3	94	94	4	-7	0	120	116	4	-11	-1	103	111
3	9	-7	59	64	3	-1	-1	777	848	4	0	4	116	84	4	-8	0	101	-68	4	-12	-1	-60	70
3	-1	0	450	485	3	-2	-1	826	909	4	1	4	-70	43	4	-9	0	153	157	4	-1	-2	456	574
3	-2	0	349	481	3	-3	-1	668	677	4	2	4	180	169	4	-10	0	180	155	4	-2	-2	419	446
3	-3	0	365	379	3	-4	-1	122	114	4	3	4	309	277	4	-11	0	89	81	4	-3	-2	-129	-29
3	-4	0	399	376	3	-5	-1	408	380	4	4	4	180	173	4	-12	0	39	49	4	-4	-2	-58	29
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3	-6	0	320	313	3	-7	-1	296	300	4	6	4	169	160	4	-2	-2	462	509	4	-6	-2	363	358
3	-7	0	330	316	3	-8	-1	268	245	4	7	4	231	229	4	-3	1	701	763	4	-7	-2	-84	-25
3	-8	0	389	393	3	-9	-1	148	142	4	8	4	128	131	4	-4	1	415	445	4	-8	-2	-89	68
3	-9	0	235	222	3	-10	-1	120	130	4	9	5	199	172	4	-5	1	242	223	4	-9	-2	171	162
3	10	0	97	99	3	-11	-1	91	86	4	0	5	213	202	4	-6	1	513	486	4	-10	-2	285	245
3	11	0	58	41	3	-12	-1	58	29	4	1	6	286	251	4	-7	1	331	325	4	-11	-2	171	124
3	-12	0	56	60	3	-1	-2	890	931	4	2	5	191	170	4	-8	1	-84	12	4	-12	-2	71	70
3	-1	1	193	224	3	-2	-2	524	544	4	3	5	162	139	4	-9	1	99	-72	4	-1	-3	296	316
3	-2	1	597	661	3	-3	-2	57	8	4	4	5	183	154	4	-10	1	179	149	4	-2	-3	84	-58
3	-3	1	303	296	3	-4	-2	361	373	4	5	5	143	136	4	-11	1	176	179	4	-3	-3	-56	16
3	-4	1	269	258	3	-5	-2	378	408	4	6	5	169	160	4	-12	1	87	82	4	-4	-3	184	187
3	-5	1	370	377	3	-6	-2	99	96	4	0	6	122	107	4	-1	2	160	-152	4	-5	-3	380	373
3	-6	1	464	509	3	-7	-2	158	-114	4	1	6	-90	-84	4	-2	2	419	424	4	-6	-3	128	140
3	-7	1	349	357	3	-8	-2	245	231	4	2	6	235	224	4	-3	2	585	572	4	-7	-3	-87	54
3	-8	1	253	233	3	-9	-2	189	195	4	3	6	331	267	4	-4	2	528	562	4	-8	-3	199	188
3	-9	1	182	214	3	-10	-2	98	81	4	4	6	187	189	4	-5	2	268	271	4	-9	-3	337	337
3	-10	1	199	204	3	-11	-2	-85	10	4	5	6	-82	36	4	-6	2	293	254	4	-10	-3	176	160
3	-11	1	146	131	3	-12	-2	112	106	4	6	6	109	101	4	-7	2	270	268	4	-11	-3	-67	56
3	-12	1	-74	48	3	-1	-3	101	76	4	7	7	275	-14	4	-8	2	347	336	4	-1	-4	78	49
3	-1	2	156	-117	3	-2	-3	102	112	4	1	7	275	219	4	-9	2	182	164	4	-2	-4	61	76
3	-2	2	349	350	3	-3	-3	338	350	4	2	7	330	340	4	-10	2	310	285	4	-3	-4	329	319
3	-3	2	273	308	3	-4	-3	409	394	4	3	7	172	151	4	-11	2	155	154	4	-4	-4	488	494
3	-4	2	268	273	3	-5	-3	100	93	4	4	7	-73	8	4	-12	2	131	131	4	-5	-4	207	192
3	-5	2	171	130	3	-6	-3	-85	27	4	5	7	117	132	4	-1	3	455	472	4	-6	-4	114	129
3	-6	2	466	461	3	-7	-3	183	184	4	6	8	184	163	4	-2	3	156	161	4	-7	-4	252	243
3	-7	2	562	581	3	-8	-3	268	245	4	7	8	139	190	4	-3	3	268	271	4	-8	-4	294	266
3	-8	2	298	292	3	-9	-3	101	104	4	8	8	83	88	4	-4	3	399	382	4	-9	-4	133	127
3	-9	2	-91	22	3	-10	-3	-95	-8	4	9	-1	96	75	4	-5	3	436	435					

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)
5	7	1	82	85	5	4	-4	237	250	5	-3	5	110	100	6	0	0	72	71	6	0	-9	-67	30
5	8	1	100	97	5	5	-4	108	106	5	-4	5	168	176	7	0	5	330	307	1	0	-10	-87	52
5	9	1	93	83	5	6	-4	-64	51	5	-5	5	302	295	8	0	0	220	233	2	0	-10	143	165
5	0	2	692	704	5	7	-4	108	97	5	-6	5	308	293	9	0	0	70	73	3	0	-10	117	152
5	1	2	357	353	5	8	-4	76	70	5	-7	5	92	86	10	0	0	-92	23	6	1	0	266	224
5	2	2	72	74	5	9	-4	57	57	5	-8	5	123	113	6	0	1	464	471	6	2	0	242	201
5	3	2	204	117	5	0	-5	135	128	5	-9	5	208	202	7	0	1	209	239	6	3	0	-66	31
5	4	2	377	393	5	1	-5	230	221	5	-10	5	176	188	8	0	1	109	97	6	4	0	68	55
5	5	2	181	140	5	2	-5	238	210	5	-11	5	79	88	9	0	1	154	138	6	5	0	169	172
5	6	2	-67	-13	5	3	-5	224	237	5	-1	6	103	106	10	0	1	-85	54	6	6	0	208	217
5	7	2	-67	64	5	4	-5	106	76	5	-2	6	242	236	6	0	2	236	236	6	7	0	140	145
5	8	2	135	142	5	5	-5	104	104	5	-3	6	154	160	7	0	2	-105	-65	6	8	0	145	148
5	9	2	87	96	5	6	-5	112	115	5	-4	6	-69	35	8	0	2	176	165	6	9	0	97	103
5	0	3	271	259	5	7	-5	112	113	5	-5	6	121	106	9	0	2	195	197	7	1	0	220	231
5	1	3	-49	8	5	8	-5	70	71	5	-6	6	246	208	10	0	2	-75	66	7	2	0	-67	71
5	2	3	239	226	5	9	-5	-45	1	5	-7	6	234	216	6	0	3	159	141	7	3	0	127	147
5	3	3	293	294	5	2	-6	107	122	5	-8	6	113	97	7	0	3	122	126	7	4	0	141	146
5	4	3	125	115	5	3	-6	241	228	5	-9	6	101	103	8	0	3	-112	83	7	5	0	155	161
5	5	3	-67	44	5	4	-6	321	314	5	-10	6	145	165	9	0	3	-93	66	7	6	0	-50	0
5	6	3	68	56	5	5	-6	175	150	5	-1	7	-68	33	10	0	3	89	113	7	7	0	-61	1
5	7	3	147	151	5	6	-6	-67	50	5	-2	7	91	86	6	0	4	249	240	7	8	0	137	152
5	8	3	93	127	5	7	-6	79	79	5	-3	7	150	148	7	0	4	-114	86	8	1	0	137	152
5	0	4	224	223	5	8	-6	65	65	5	-4	7	112	110	8	0	4	-105	40	8	2	0	142	133
5	1	4	219	191	5	9	-6	39	42	5	-5	7	-65	4	9	0	4	136	141	8	3	0	217	223
5	2	4	128	145	5	4	-7	216	205	5	-6	7	-61	43	6	0	5	-118	54	8	4	0	186	174
5	3	4	146	184	5	5	-7	255	252	5	-7	7	123	127	7	0	5	136	124	7	5	0	171	148
5	4	4	76	68	5	6	-7	134	135	5	-8	7	124	134	8	0	5	133	140	8	6	0	-38	35
5	5	4	104	97	5	7	-7	-49	41	5	-1	8	131	138	6	0	6	156	139	8	7	0	79	88
5	6	4	133	139	5	-1	0	220	255	5	-2	8	-54	57	7	0	6	202	205	9	1	0	79	77
5	7	4	59	45	5	-2	0	262	306	5	-3	8	198	94	6	0	7	156	161	9	2	0	170	157
5	8	4	33	37	5	-3	0	300	408	5	-4	8	79	79	6	0	8	248	250	10	3	0	142	145
5	9	4	135	126	5	-4	0	76	76	5	-5	8	-48	39	7	0	1	253	233	9	4	0	78	76
5	1	5	-63	-14	5	-5	0	104	104	5	-6	8	63	70	8	0	1	319	260	9	5	0	45	57
5	2	5	81	82	5	-6	0	150	146	5	-1	-1	39	40	9	0	1	159	141	10	1	0	95	114
5	3	5	169	162	5	-7	0	184	206	5	-2	-1	99	91	10	0	1	-95	74	10	2	0	190	100
5	4	5	125	121	5	-8	0	-65	16	5	-3	-1	239	232	6	0	2	354	311	10	3	0	171	148
5	5	5	64	65	5	-9	0	161	145	5	-4	-1	273	252	7	0	2	-333	332	1	-1	0	680	707
5	6	5	74	83	5	-10	0	183	179	5	-5	-1	92	90	8	0	2	-110	121	6	-1	0	65	53
5	7	5	92	103	5	-11	0	244	225	5	-6	-1	187	192	9	0	2	140	112	6	-2	0	179	180
5	8	5	-59	-32	5	-12	0	108	102	5	-7	-1	210	215	10	0	2	172	148	6	-3	0	391	407
5	9	5	-10	37	5	-1	1	113	109	5	-8	0	290	250	10	0	3	202	102	7	-3	0	177	148
5	0	6	200	175	5	-2	1	246	245	5	-9	-1	201	174	6	0	3	117	126	6	-4	0	76	83
5	1	6	135	124	5	-3	1	403	406	5	-10	-1	241	220	7	0	3	254	237	6	-6	0	246	234
5	2	6	-60	57	5	-4	1	356	367	5	-11	-1	157	143	8	0	3	205	183	6	-7	0	468	450
5	3	6	84	83	5	-5	1	-53	53	5	-12	-1	116	112	9	0	3	142	142	6	-8	0	256	263
5	4	6	128	117	5	-6	1	135	-108	5	-1	2	430	426	6	0	4	181	181	9	-9	0	127	133
5	5	6	224	210	5	-7	1	185	163	5	-2	2	420	437	1	0	4	314	306	6	-10	0	119	111
5	6	6	99	87	5	-8	1	377	362	5	-3	2	340	336	6	0	4	369	377	6	-11	0	144	147
5	7	6	-51	-6	5	-9	1	97	82	5	-4	2	201	188	7	0	4	-113	-56	6	-12	0	91	126
5	8	6	77	70	5	-10	1	-68	14	5	-5	2	239	241	8	0	4	-118	9	7	-1	0	142	145
5	9	6	146	146	5	-11	1	153	144	5	-6	2	351	338	9	0	4	182	182	7	-2	0	44	-12
5	0	7	200	175	5	-12	1	148	156	5	-7	2	316	307	10	0	4	132	132	7	-3	0	269	261
5	1	7	135	124	5	-1	2	433	469	5	-8	2	244	234	1	0	5	-88	-12	7	-4	0	485	492
5	2	7	416	414	5	-2	2	251	250	5	-9	2	161	169	2	0	5	265	261	7	-5	0	279	279
5	3	7	232	211	5	-3	2	227	219	5	-10	2	122	136	3	0	5	301	305	7	-6	0	142	153
5	4	7	141	145	5	-4	2	393	419	5	-11	2	112	126	4	0	5	257	246	7	-7	0	201	188
5	5	7	278	273	5	-5	2	213	194	5	-1	3	424	456	6	0	5	201	198	7	-8	0	260	251
5	6	7	455	502	5	-6	2	-59	-1	5	-2	3	407	418	7	0	5	-122	72	7	-9	0	143	130
5	7	7	194	151	5	-7	2	-62	56	5	-3	3	145	130	8	0	5	-120	34	7	-10	0	-67	52
5	8	7	78	54	5	-8	2	254	220	5	-4	3	167	148	9	0	5	152	137	7	-11	0	-31	31
5	9	7	131	123	5	-9	2	170	182	5	-5	3	412	397	10	0	5	66	82	8	-1	0	328	343
5	0	8	180	179	5	-10	2	75	74	5	-6	3	354	332	1	0	6	345	359	8	-2	0	714	225
5	1	8	131	102	5	-11	2	-57	36	5	-7	3	149	164	2	0	6	112	-85	8	-3	0	72	83
5	2	8	-231	-220	5	-12	2	77	76	5	-8	3	115	110	3	0	6	-103	-71	8	-4	0	193	202
5	3	8	181	177	5	-1	3	599	630	5	-9	3	243	204	4	0	6	216	224	8	-5	0	224	216
5	4	8	419	405	5	-2	3	341	351	5	-10	3	210	204	5	0	6	359	356	8	-6	0	46	81
5	5	8	129	121	5	-3	3	109	106	5	-11	3	79	80	6	0	6	-190	181	8	-7	0	61	-29
5	6	8	-55	-7	5	-4	3	368	385	5	-1	4	242	235	7	0	6	-123	86	8	-8	0	84	91
5	7	8	147	191	5	-5	3	393	388	5	-2	4	256	265	8	0	6	144	117	8	-9	0	178	127
5	8	8	286	309	5	-6	3	248	228	5	-3	4	287	259	9	0	6	124	109	8	-10	0		

Table 5. Bond lengths (Å) and angles (°). Standard deviations are given in parentheses.

TeS ₄ coordination group			
Te —S(1)	= 2.691(4)	∠S(1) —Te —S(2)	= 88.85(9)
Te —S(2)	= 2.668(3)		
Benzenethiosulphonate group			
S(2) —S(3)	= 2.018(4)	∠Te —S(2) —S(3)	= 105.73(13)
S(3) —O(1)	= 1.443(9)	∠S(2) —S(3) —O(1)	= 107.2(4)
S(3) —O(2)	= 1.443(9)	∠S(2) —S(3) —O(2)	= 111.7(4)
S(3) —C(5)	= 1.777(11)	∠S(2) —S(3) —C(5)	= 105.7(4)
C(5) —C(6)	= 1.364(16)	∠O(1) —S(3) —O(2)	= 116.7(6)
C(6) —C(7)	= 1.412(18)	∠O(1) —S(3) —C(5)	= 108.3(5)
C(7) —C(8)	= 1.386(18)	∠O(2) —S(3) —C(5)	= 106.7(5)
C(8) —C(9)	= 1.390(18)	∠S(3) —C(5) —C(6)	= 119.1(8)
C(9) —C(10)	= 1.401(17)	∠S(3) —C(5) —C(10)	= 117.0(9)
C(10) —C(5)	= 1.402(14)	∠C(10) —C(5) —C(6)	= 123.9(10)
		∠C(5) —C(6) —C(7)	= 117.7(10)
		∠C(6) —C(7) —C(8)	= 120.6(13)
		∠C(7) —C(8) —C(9)	= 120.0(13)
		∠C(8) —C(9) —C(10)	= 120.9(10)
		∠C(9) —C(10) —C(5)	= 117.0(10)
Trimethylenethiourea group			
S(1) —C(1)	= 1.716(11)	∠Te —S(1) —C(1)	= 103.9(4)
C(1) —N(1)	= 1.352(15)	∠S(1) —C(1) —N(1)	= 117.0(8)
C(1) —N(2)	= 1.313(13)	∠S(1) —C(1) —N(2)	= 123.0(8)
N(1) —C(4)	= 1.469(17)	∠N(1) —C(1) —N(2)	= 119.9(10)
N(2) —C(2)	= 1.483(16)	∠C(1) —N(1) —C(4)	= 122.5(10)
C(2) —C(3)	= 1.532(17)	∠C(1) —N(2) —C(2)	= 123.8(10)
C(4) —C(3)	= 1.537(19)	∠N(1) —C(4) —C(3)	= 110.5(11)
		∠N(2) —C(2) —C(3)	= 109.0(9)
		∠C(2) —C(3) —C(4)	= 106.9(10)

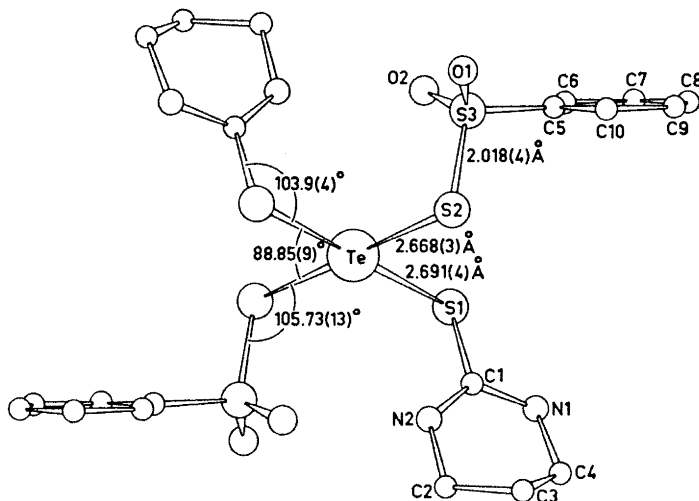


Fig. 1. The molecule as seen along the normal to a plane through Te, O(2) and the midpoint between S(1) and S(2).

With tellurium in a centre of symmetry, the TeS_4 group is exactly planar. The S—Te—S angle deviates slightly from 90° , presumably because of hydrogen bonding or crystal packing effects.

In uncomplexed tellurium dibenzenethiosulphonate,⁸ the Te—S bonds are 2.41(3) Å, and the S—Te—S angle $100(2)^\circ$. The bond length, 2.41 Å, agrees with the sum of the single covalent radii for tellurium and sulphur,⁹ 1.37 Å and 1.04 Å, respectively. In the complex, the Te—S bonds are longer, 2.691(4) Å and 2.668(3) Å. With single covalent radius for sulphur, the Te—S bond lengths conform to a bonding radius of 1.64 Å for tellurium(II) in centrosymmetric, square-planar complexes, as proposed by Foss.^{10,11} The dimensions of the TeS_4 group are in accord with a three-centre two-electron-pair bonding scheme, based on tellurium $5p$ orbitals.

The S—S bond in the benzenethiosulphonate group, 2.018(4) Å, is to be compared with 2.08(4) Å for the S—S bond in uncomplexed tellurium dibenzenethiosulphonate,⁶ and 1.98(1) Å for the S—S bond in ionic sodium methanethiosulphonate monohydrate.¹² The short S—S bond in the complex relative to the covalent compound, indicates a covalency lower than one for the Te—S bond in the complex, and hence is in agreement with the large Te—S distances found.

The S—O bonds of the benzenethiosulphonate group are both equal to 1.443(9) Å, while the S—C bond is 1.777(11) Å. The arrangement of atoms bonded to S(3) is approximately tetrahedral, with greatest deviation in the O—S—O angle, which is $116.7(6)^\circ$. The benzene ring has a somewhat large angle at the carbon atom bonded to sulphur. This C(10)—C(5)—C(6) angle is $123.9(10)^\circ$. The atoms of a least squares plane through S(3) and the benzene ring do not deviate more than 0.008 Å from this plane.

Bond lengths and angles of the trimethylenethiourea group do not deviate significantly neither from the values found by Dias and Truter¹³ for trimethylenethiourea, nor from the values found by Luth and Truter¹⁴ for trimethylenethiourea as a ligand in dichloro-tetrakis(trimethylenethiourea)nickel(II). Excluding C(3), a least squares plane through the trimethylenethiourea group shows an approximately planar group with maximum deviation for C(1), which is 0.023 Å from the plane. C(3) is 0.683 Å from this plane, at the same side as C(1). The plane makes an angle of 89.7° with the TeS_4 plane.

Each of the hydrogen atoms bonded to the trimethylenethiourea nitrogen atoms appear to be engaged in hydrogen bonding to the benzenethiosulphonate oxygen atoms. The N(1)⋯O(1') distance, where O(1') is at $x-1, y, z$ relative to O(1), is 2.942(13) Å. The C(1)—N(1)⋯O(1') angle is $139.1(8)^\circ$, and the C(4)—N(1)⋯O(1') angle is $97.8(7)^\circ$. This bond occurs between neighbouring molecules. The N(2)⋯O(2') distance, where O(2') is at $-x, -y, -z$ relative to O(2), is 2.864(12) Å. The C(1)—N(2)⋯O(2') angle is $132.0(8)^\circ$, and the C(2)—N(2)⋯O(2') angle is $101.0(7)^\circ$. This bond occurs within the molecule. O(1') and O(2') are 0.459 Å and 0.829 Å, respectively, out of the least squares plane referred to above, through the trimethylenethiourea group, and they are both on the opposite side of C(3). These dimensions are in the range found for N—H⋯O hydrogen bonds in other compounds.¹⁵

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