ACTA CHEMICA SCANDINAVICA 25 (1971) 838–846

A Refinement of the Crystal Structure of Tellurium Dibenzenethiosulphonate

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The crystal structure of tellurium dibenzenethiosulphonate, $Te(S_2O_2C_6H_5)_2$, has been refined by full-matrix least squares methods, using three-dimensional diffractometer data, to a conventional R value of 0.038. The crystals are orthorhombic, space group *Pbcn* (No. 60), with $a = 14.467$ Å, $b = 11.137$ Å, $c = 10.455$ Å, and four molecules in the unit cell.

The tellurium atom of the nonplanar S–S–Te–S–S chain lies on a two-fold axis, and the molecule thus occurs in a *trans* form. The following dimensions of the chain, with standard deviations in parentheses, have been found: Te–S = 2.380(2) Å, S–S = 2.080(2) Å, ∠S–Te–S = 97.61(6)°, ∠S–S–Te = 103.46(7)°. The dihedral angle between the S–S–Te and S–Te–S planes is 97.4°.

The crystal structure of a *trans* square-planar complex of tellurium dibenzenethiosulphonate with trimethylenethiourea [1] was recently reported, and the structures of two similar complexes, one with ethylenethiourea and another with tetramethylthiourea, are under study in this Laboratory. In connection with investigations of this type of complex, it was thought of interest to refine the structure of uncomplexed tellurium dibenzenethiosulphonate, which was reported in 1956 by Øyum and Foss.[2] With more modern facilities, it should be possible to determine the structural parameters more accurately. The lengths of the Te-S and S–S bonds are of special interest for comparison with the corresponding bond lengths in the complexes mentioned above.

EXPERIMENTAL

The yellow orthorhombic crystals of $Te(S_2O_2C_6H_5)_2$ were prepared by Foss.[3]

The data collection was made by means of a Siemens automatic, off-line, single crystal diffractometer (AED), using (Ni-filtered) Cu$K\alpha$ radiation. The diffractometer was operated as a three-circle instrument.

A bipyramidal crystal with the following dimensions, given as distances from a common origin to the faces, was used: distances to $(\bar{1}00)$ and $(100) = 0.046$ mm; to $(\bar{1}\bar{1}1)$ and $(11\bar{1}) = 0.061$ mm; to $(\bar{1}1\bar{1})$ and $(1\bar{1}1) = 0.062$ mm; to $(\bar{1}11)$ and $(1\bar{1}\bar{1}) = 0.056$ mm; and to $(\bar{1}\bar{1}\bar{1})$ and $(111) = 0.059$ mm.

The crystal was mounted with the a axis approximately along the ϕ axis of the diffractometer. Its orientation and cell dimensions were determined by measuring θ, χ, and ϕ for three non-coplanar reciprocal vectors, and the setting angles for all reflections could be calculated.

For determination of unit cell dimensions by least squares, θ angles of 8 reflections at high θ were measured.

Intensity data were collected using a scintillation counter and $\theta - 2\theta$ scan technique. The scan-speed was chosen to be 0.5 degrees per minute, with automatic choice of greater scan-speed for strong reflections. When an attenuation filter was needed to avoid counting losses, the proper one was automatically inserted into the primary beam.

Each reflection was scanned between $\theta_1 = \theta - 0.40°$, and $\theta_2 = \theta + 0.40° + 0.15°\mathrm{tg}\theta$, where θ is the Bragg angle for the α_1 peak. The scan was performed by going from θ to θ_1, then from θ_1 to θ_2, and finally from θ_2 to θ. The intensities over each scanning range were recorded at the end of each step. The background was measured for one half of the total scan time at θ_1, and one half at θ_2. This scan procedure provides a check of the setting angles.

Two reference reflections were measured at intervals of 50 reflections, and the setting angles of these two reflections were checked by horizontal and vertical half shutters.

The net intensities were brought to a common scale by using the intensity variations of the reference reflections. The lower limit for observed reflections was set equal to two times the standard deviation in net intensity. The standard deviation is $(I_t + I_b)^{\frac{1}{2}}$, where I_t is the total intensity, and I_b is the background intensity. Out of 1631 reflections with $\theta < 70°$, 1373 were found stronger than the lower limit. The remaining 258 reflections were assigned an intensity equal to the limit, and labelled as unobserved reflections.

Absorption ($\mu = 193$ cm^{-1}), Lorentz, and polarization corrections were carried out. A modified version of the absorption correction method described by Busing and Levy [4] was applied, using a $6 \times 8 \times 10$ grid.

The calculated structure factors were based on the scattering curve of Stewart *et al.*[5] for hydrogen, and the curves listed in *International Tables* [6] for all other atoms. Using the $\Delta f'$ and $\Delta f''$ values given by Cromer,[7] the tellurium and sulphur scattering 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

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

Table 1. Computer programs.

Name	Function	Origin
CELL BDLS DIAN	Cell dimensions Least squares refinement Distances and angles	Weizmann Institute of Science, Rehovoth, Israel
ABCD	Absorption correction	As above. Modified by the present author, to take into account a general orientation of the crystal
AED1	Setting angles for the diffractometer	Mr. Knut Maartman-Moe, Chemical Institute, University of Bergen, Norway
PLAN	Weighted least squares planes	
DAT1	Data processing	Present author
ASEN	Fourier summation	
ZACH	Extinction correction	

where K is a scale factor, and the weight, W, is the inverse of the variance of F_o. The variance of F_o is

$$\sigma^2(F_o) = F_o^2[I_t + I_b + k^2(I_t - I_b)^2]/4(I_t - I_b)^2$$

where k may be interpreted as the relative standard deviation in the scaling curve based on reference reflections. It was given the value 0.008. Non-observed reflections for which $K|F_c|$ is greater than 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. The programs used are listed in Table 1.

CRYSTAL DATA

The crystals of tellurium dibenzenethiosulphonate are orthorhombic. Based on 8 high-angle θ-values measured on the diffractometer, a least squares procedure gave the following cell dimensions, with standard deviations in parentheses:

$a = 14.467(1)$ Å; $b = 11.137(1)$ Å; $c = 10.455(1)$ Å; $V = 1684.5(3)$ Å^3.

Other pertinent values from the earlier study of Øyum and Foss [2] are

$$M = 474.07;\ F(000) = 1020;\ Z = 4.$$

Systematic absences are $hk0$ when $h+k$ is odd, $h0l$ when l is odd, $0kl$ when k is odd. The uniquely determined space group is *Pbcn* (No. 60).

REFINEMENT OF THE STRUCTURE

With four molecules in the unit cell, a two-fold rotation axis must pass through the tellurium atom. Using the positional and thermal parameters given by Øyum and Foss [2] for the non-hydrogen atoms, refinement by least squares was started. Refinement on the scale factor, positional parameters and individual thermal parameters resulted in an R value of 0.065. Introduction of anisotropic thermal parameters for the tellurium and sulphur atoms lowered the R value to 0.051, and introduction of anisotropic thermal parameters for the rest of the atoms lowered it to 0.040. Applying the test of Hamilton,[8] both changes in the model were found to be significant at the 0.5 % level.

There seemed to be a marked tendency of $|F_o|$ values for strong, low-order reflections to be smaller than the corresponding $K|F_c|$ values. A correction for extinction was therefore carried out, using the expression given by Zachariasen.[9] The absorption term in this expression was set equal to 1. With I_o on an absolute scale, the value of the extinction parameter, C, was found to be 4.7×10^{-7}. Additional refinement on the parameters mentioned above lowered the R value to 0.038.

A three-dimensional difference Fourier summation at this point showed peaks between 0.40 and 0.67 e/Å^3 at the positions where the hydrogen atoms would be expected. The hydrogen positions were taken from the difference map, and a refinement with hydrogen included was attempted. This attempt was not successful, however, as the hydrogen atoms were shifted to

unreasonable positions, with C–H bond lengths between 0.81 and 1.12 Å, and one of the C–C–H bond angles as low as 99°. The hydrogen atoms were therefore left out of the structure factor calculations and the refinement.

In a final full-matrix, least squares cycle no shift was greater than 0.4 times the standard deviation. The final R value, with non-observed reflections included when $K|F_c|$ exceeds the observable limit, is 0.038. The final atomic coordinates are listed in Table 2, and the thermal parameters in Table 3. The structure factors based on the final parameters are listed in Table 4.

Table 2. Atomic coordinates in fractions of cell edges. Standard deviations from the least squares refinement in parentheses.

	x	y	z
Te	0.0	0.04397 (5)	0.25
S(2)	−0.06880 (10)	0.18473 (14)	0.39244 (16)
S(1)	0.03909 (11)	0.23095 (13)	0.51441 (15)
O(1)	−0.0035 (4)	0.3132 (4)	0.6018 (5)
O(2)	0.0832 (4)	0.1246 (4)	0.5619 (5)
C(1)	0.1187 (4)	0.3095 (5)	0.4153 (6)
C(2)	0.2036 (5)	0.2556 (6)	0.3895 (8)
C(3)	0.2623 (5)	0.3167 (7)	0.3050 (9)
C(4)	0.2353 (5)	0.4259 (7)	0.2530 (10)
C(5)	0.1503 (5)	0.4778 (6)	0.2851 (8)
C(6)	0.0909 (5)	0.4203 (6)	0.3679 (7)

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. Standard deviations in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Te	426 (3)	396 (3)	402 (3)	0	0	4 (3)
S(2)	391 (7)	453 (8)	442 (9)	38 (6)	− 14 (7)	68 (7)
S(1)	615 (8)	428 (7)	343 (8)	− 25 (7)	10 (7)	10 (7)
O(1)	1063 (35)	625 (26)	469 (27)	− 39 (33)	−151 (22)	189 (33)
O(2)	751 (29)	551 (25)	574 (30)	− 19 (23)	182 (24)	−129 (25)
C(1)	493 (31)	412 (28)	436 (33)	− 65 (25)	− 20 (27)	− 32 (27)
C(2)	514 (34)	554 (37)	670 (47)	38 (30)	− 31 (37)	− 56 (34)
C(3)	490 (37)	700 (49)	909 (58)	− 57 (34)	82 (42)	34 (37)
C(4)	596 (37)	746 (46)	900 (54)	−199 (32)	− 21 (59)	49 (50)
C(5)	641 (38)	509 (41)	789 (64)	−115 (30)	28 (34)	66 (36)
C(6)	669 (38)	387 (29)	589 (44)	− 39 (27)	30 (31)	− 9 (33)

RESULTS

The tellurium dibenzenethiosulphonate molecule has been described by Øyum and Foss.[2] Here the new results will be recorded, and the differences, although not great, between the present investigation and the older one, will be pointed out.

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. A drawing of the molecule, with the most important bond lengths and angles, is presented in Fig. 1.

Table 4. 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)	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	0	C	3432	3514	4	12	0	348	-343	6	6	1	758	-793	7	0	2	313	-311	1	6	2	226	-220
4	0	C	2240	2246	6	12	0	401	-399	7	6	1	352	351	8	0	2	1590	-1593	2	6	2	149	-138
6	0	C	345	347	8	12	0	326	-315	8	6	1	585	-577	9	0	2	368	354	3	6	2	388	-368
8	0	C	758	766	1	13	0	270	-271	9	6	1	325	329	10	0	2	886	-894	4	6	2	66	-36
10	0	C	598	599	3	13	0	205	-206	10	6	1	666	-668	11	0	2	512	507	5	6	2	284	-285
12	0	C	748	740	5	13	0	155	-144	11	6	1	196	199	12	0	2	974	-963	6	6	2	85	-68
14	0	0	628	651	1	1	1	909	-954	12	6	1	583	-576	13	0	2	186	176	7	6	2	-56	-8
16	0	C	444	451	2	1	1	788	802	13	6	1	57	64	14	0	2	611	-596	8	6	2	135	138
1	1	0	1752	1852	3	1	1	69	25	14	6	1	461	-453	15	0	2	52	-51	9	6	2	417	407
3	1	C	2143	2253	4	1	1	202	227	15	6	1	-67	-52	16	0	2	398	-381	10	6	2	220	211
5	1	0	1117	1160	5	1	1	94	46	1	7	1	1199	-1229	17	0	2	133	-130	11	6	2	189	185
7	1	C	567	656	6	1	1	310	298	2	7	1	479	-502	1	1	2	1146	-1175	12	6	2	181	199
9	1	C	651	674	7	1	1	173	127	3	7	1	848	-860	2	1	2	1077	-1061	13	6	2	84	99
11	1	0	784	753	8	1	1	279	303	4	7	1	613	-606	3	1	2	1863	-1848	14	6	2	130	132
13	1	C	607	602	9	1	1	99	-83	5	7	1	506	-492	4	1	2	705	-676	15	6	2	-70	-84
15	1	C	525	521	10	1	1	504	489	6	7	1	536	-554	5	1	2	1521	-1510	1	7	2	112	112
17	1	0	323	327	11	1	1	225	-228	7	7	1	560	-568	6	1	2	-52	1	2	7	2	183	179
0	2	C	785	-788	12	1	1	354	343	8	7	1	-57	-8	7	1	2	1257	-1301	3	7	2	-58	39
2	2	0	252	86	13	1	1	240	-223	9	7	1	355	-350	8	1	2	191	194	4	7	2	167	165
4	2	C	426	440	14	1	1	59	-53	10	7	1	145	145	9	1	2	1027	-1026	5	7	2	112	121
6	2	0	1303	1379	15	1	1	290	-292	11	7	1	456	-449	10	1	2	227	235	6	7	2	71	73
8	2	0	1346	1367	16	1	1	128	-132	12	7	1	103	122	11	1	2	799	-788	7	7	2	361	363
10	2	C	885	911	17	1	1	65	-104	13	7	1	444	-443	12	1	2	-57	87	8	7	2	-58	-32
12	2	0	756	820	0	2	1	379	-394	14	7	1	71	15	13	1	2	467	-452	9	7	2	309	326
14	2	C	432	424	1	2	1	311	-346	15	7	1	353	-356	14	1	2	64	-17	10	7	2	71	-15
16	2	0	205	202	2	2	1	1654	-1793	0	8	1	768	-790	15	1	2	429	-419	11	7	2	334	327
1	3	C	124	134	3	2	1	191	-161	1	8	1	-61	-69	16	1	2	76	-71	12	7	2	-51	-17
3	3	C	1118	1152	4	2	1	781	-854	2	8	1	725	-736	17	1	2	303	-304	13	7	2	217	225
5	3	C	1379	1483	5	2	1	-49	-21	3	8	1	211	-213	0	2	2	3137	-3191	14	7	2	-64	-42
7	3	C	1655	1721	6	2	1	966	-1002	4	8	1	664	-687	1	2	2	114	-71	0	8	2	766	764
9	3	0	1268	1216	7	2	1	191	192	5	8	1	270	-278	2	2	2	2182	-2276	1	8	2	-62	47
11	3	C	666	650	8	2	1	236	-245	6	8	1	422	-425	3	2	2	1027	996	2	8	2	551	532
13	3	0	120	65	9	2	1	140	140	7	8	1	258	-251	4	2	2	1489	-1504	3	8	2	102	96
15	3	0	190	176	10	2	1	452	-466	8	8	1	456	-458	5	2	2	459	440	4	8	2	676	679
17	3	C	172	168	11	2	1	190	183	9	8	1	161	-147	6	2	2	1156	-1226	5	8	2	162	173
0	4	0	1265	1267	12	2	1	337	-350	10	8	1	453	-440	7	2	2	187	159	6	8	2	327	315
2	4	0	817	843	13	2	1	107	113	11	8	1	-51	-6	8	2	2	755	-732	7	8	2	167	167
4	4	C	1008	1022	14	2	1	426	-417	12	8	1	321	-317	9	2	2	156	-157	8	8	2	335	323
6	4	C	1109	1114	15	2	1	-50	4	13	8	1	-44	11	10	2	2	406	-398	9	8	2	219	-199
8	4	C	297	323	16	2	1	266	-264	14	8	1	229	-226	11	2	2	366	-373	10	8	2	217	220
10	4	C	102	54	17	2	1	174	-92	1	9	1	266	-264	12	2	2	329	-306	11	8	2	208	-195
12	4	C	143	-115	1	3	1	1268	-1335	2	9	1	231	224	13	2	2	65	-66	12	8	2	189	188
14	4	C	115	100	2	3	1	50	20	3	9	1	346	-354	14	2	2	329	-315	13	8	2	152	-155
16	4	0	-72	72	3	3	1	1159	-1190	4	9	1	290	279	15	2	2	-49	56	14	8	2	95	82
1	5	C	606	617	4	3	1	114	87	5	9	1	653	-664	16	2	2	241	-234	1	9	2	895	907
3	5	C	573	602	5	3	1	1092	-1114	6	9	1	361	352	17	2	2	-68	63	2	9	2	82	73
5	5	C	74	57	6	3	1	543	-549	7	9	1	565	-560	1	3	2	1291	-1341	3	9	2	759	742
7	5	C	600	-572	7	3	1	952	-993	8	9	1	87	61	2	3	2	855	909	4	9	2	136	125
9	5	0	262	-262	8	3	1	517	-522	9	9	1	361	-356	3	3	2	1459	-1465	5	9	2	548	537
11	5	C	61	-65	9	3	1	776	-800	10	9	1	93	-98	4	3	2	1007	973	6	9	2	-60	-4
13	5	0	212	203	10	3	1	417	-431	11	9	1	219	-207	5	3	2	676	-682	7	9	2	442	435
15	5	C	91	92	11	3	1	480	-472	12	9	1	98	-109	6	3	2	199	230	8	9	2	-57	-62
0	6	C	59	-46	12	3	1	131	-139	13	9	1	152	-145	7	3	2	372	-404	9	9	2	288	280
2	6	C	80	24	13	3	1	461	-460	0	10	1	73	-76	8	3	2	211	-212	10	9	2	103	-114
4	6	C	358	-371	14	3	1	135	127	1	10	1	205	222	9	3	2	364	-350	11	9	2	200	195
6	6	0	226	-159	15	3	1	155	-154	2	10	1	77	-44	10	3	2	345	-327	12	9	2	-43	-47
8	6	C	81	-48	16	3	1	91	93	3	10	1	476	480	11	3	2	471	-491	13	9	2	123	130
10	6	C	97	-88	17	3	1	205	-217	4	10	1	272	-268	12	3	2	173	-173	0	10	2	508	499
12	6	C	213	207	0	4	1	469	-492	5	10	1	395	395	13	3	2	301	-290	1	10	2	-64	-41
14	6	C	209	213	1	4	1	295	313	6	10	1	150	-156	14	3	2	88	-99	2	10	2	493	514
1	7	C	508	-525	2	4	1	706	-772	7	10	1	105	107	15	3	2	293	-295	3	10	2	-62	-44
3	7	C	632	-619	3	4	1	239	241	8	10	1	320	-310	16	3	2	174	113	4	10	2	427	425
5	7	C	481	-480	4	4	1	1045	-1179	9	10	1	-50	-17	17	3	2	173	-170	5	10	2	136	-122
7	7	C	293	-286	5	4	1	-49	77	10	10	1	183	-176	0	4	2	344	-317	6	10	2	426	433
9	7	C	143	100	6	4	1	1207	-1216	11	10	1	-43	-60	1	4	2	343	339	7	10	2	85	-82
11	7	C	-54	39	7	4	1	282	-275	12	10	1	89	-88	2	4	2	227	-252	8	10	2	404	390
13	7	C	-48	-40	8	4	1	1097	-1115	1	11	1	96	-62	3	4	2	67	31	9	10	2	119	116
15	7	C	112	-102	9	4	1	289	-279	2	11	1	-61	-18	4	4	2	120	8	10	10	2	380	370
0	8	C	908	-879	10	4	1	703	-719	3	11	1	-60	-35	5	4	2	65	-52	11	10	2	83	89
2	8	C	611	-612	11	4	1	271	-264	4	11	1	-58	-41	6	4	2	419	-424	1	11	2	279	267
4	8	C	476	-461	12	4	1	346	-337	5	11	1	-57	-12	7	4	2	-53	-27	2	11	2	183	-188
6	8	C	100	-99	13	4	1	168	-163	6	11	1	114	-122	8	4	2	485	-490	3	11	2	332	333
8	8	0	380	-376	14	4	1	218	-202	7	11	1	108	-114	9	4	2	183	-187	4	11	2	211	-201
10	8	C	283	-277	15	4	1	-58	7	8	11	1	-48	-24	10	4	2	544	-538	5	11	2	356	357
12	8	C	433	-432	16	4	1	226	-240	9	11	1	53	36	11	4	2	-56	-16	6	11	2	-53	5
14	8	C	320	-331	1	5	1	1353	-1422	10	11	1	-40	-2	12	4	2	488	-493	7	11	2	376	379
1	9	C	321	-322	2	5	1	397	412	0	12	1	-57	-37	13	4	2	75	-54	8	11	2	90	77
3	9	0	317	-329	3	5	1	957	-974	1	12	1	134	-133	14	4	2	374	-375	9	11	2	346	353
5	9	C	400	-389	4	5	1	404	385	2	12	1	58	-57	15	4	2	-59	60	10	11	2	110	119
7	9	C	375	-388	5	5	1	1277	-1313	3	12	1	283	-278	16	4	2	166	-173	0	12	2	401	389
9	9	C	382	-377	6	5	1	631	611	4	12	1	-52	14	1	5	2	74	-8	1	12	2	-55	53
11	9	C	401	-356	7	5	1	926	-950	5	12	1	236	-235	2	5	2	407	-414	2	12	2	425	427
13	9	C	391	-387	8	5	1	266	247	6	12	1	153	147	3	5	2	63	-12	3	12	2	-52	8
0	10	C	449	-412	9	5	1	931	-935	7	12	1	131	-125	4	5	2	339	-346	4	12	2	388	394
2	10	0	479	-472	10	5	1	88	76	8	12	1	160	168	5	5	2	118	-108	5	12	2	-48	15
4	10	C	671	-649	11	5	1	602	-592	1	13	1	65	68	6	5	2	189	-174	6	12	2	395	390
6	10	C	636	-625	12	5	1	-55	-42	2	13	1	-46	-47	7	5	2	175	-145	7	12	2	-42	-25
8	10	C	401	-389	13	5	1	514	-503	3	13	1	176	173	8	5	2	84	-81	8	12	2	267	266
10	10	C	361	-367	14	5	1	-50	-51	4	13	1	93	-94	9	5	2	469	-469	1	13	2	424	428
12	10	C	184	-189	15	5	1	283	-281	5	13	1	182	179	10	5	2	146	144	2	13	2	75	78
1	11	C	700	-699	16	5	1	-72	5	0	0	2	874	-786	11	5	2	260	-254	3	13	2	377	381
3	11	0	647	-634	0	6	1	1135	-1225	1	0	2	593	519	12	5	2	200	194	4	13	2	84	84
5	11	0	546	-535	1	6	1	293	-294	2	0	2	490	292	13	5	2	174	-178	1	1	3	268	317
7	11	C	597	-583	2	6	1	1512	-1572	3	0	2	1220	-1176	14	5	2	133	134	2	1	3	624	603
9	11	C	413	-412	3	6	1	295	-278	4	0	2	1522	-1547	15	5	2	-65	-45	3	1	3	1121	1044
0	12	C	319	-309	4	6	1	1035	-1088	5	0	2	869	-855	16	5	2	74	-70	4	1	3	975	990
2	12	C	411	-414	5	6	1	85	53	6	0	2	1379	-1377	0	6	2	169	-187	5	1	3	856	872

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)
6	1	3	688	665	13	7	3	274	257	13	2	4	-55	-40	12	9	4	-39	-39	1	6	5	174	171
7	1	3	886	864	14	7	3	99	8	14	2	4	692	683	0	10	4	729	-729	2	6	5	768	-781
8	1	3	307	256	0	8	3	406	403	15	2	4	51	-37	1	10	4	-62	-22	3	6	5	531	523
9	1	3	408	405	1	8	3	84	-72	16	2	4	417	425	2	10	4	711	-722	4	6	5	716	-714
10	1	3	-55	65	2	8	3	548	510	1	3	4	822	825	3	10	4	-59	19	5	6	5	413	399
11	1	3	217	224	3	8	3	235	-238	2	3	4	747	727	4	10	4	680	-677	6	6	5	643	-633
12	1	3	123	-115	4	8	3	644	653	3	3	4	811	797	5	10	4	-57	-43	7	6	5	170	164
13	1	3	57	51	5	8	3	292	-281	4	3	4	373	393	6	10	4	343	-356	8	6	5	608	-591
14	1	3	-52	-10	6	8	3	523	512	5	3	4	280	278	7	10	4	-52	-1	9	6	5	-57	-47
15	1	3	121	-116	7	8	3	192	-180	6	3	4	103	112	8	10	4	297	-293	10	6	5	569	-579
16	1	3	55	-62	8	8	3	499	499	7	3	4	130	91	9	10	4	60	51	11	6	5	-53	-62
17	1	3	-55	-12	9	8	3	96	-85	8	3	4	85	-71	10	10	4	233	-233	12	6	5	443	-449
0	2	3	395	400	10	8	3	382	383	9	3	4	251	237	1	11	4	700	-702	13	6	5	101	-104
1	2	3	605	595	11	8	3	97	-88	10	3	4	-58	-45	2	11	4	-56	-26	14	6	5	294	-303
2	2	3	782	772	12	8	3	314	303	11	3	4	542	529	3	11	4	587	-580	1	7	5	889	-915
3	2	3	540	530	13	8	3	-44	1	12	3	4	141	-141	4	11	4	-54	7	2	7	5	194	187
4	2	3	157	52	1	9	3	786	767	13	3	4	542	528	5	11	4	389	-383	3	7	5	665	-675
5	2	3	495	481	2	9	3	-62	-11	14	3	4	-50	-1	6	11	4	-49	25	4	7	5	86	92
6	2	3	605	608	3	9	3	362	368	15	3	4	414	417	7	11	4	181	-165	5	7	5	648	-634
7	2	3	77	-68	4	9	3	129	113	16	3	4	-69	2	8	11	4	-43	49	6	7	5	-59	51
8	2	3	605	597	5	9	3	-61	22	0	4	4	1051	1050	0	12	4	417	-419	7	7	5	550	-542
9	2	3	87	-86	6	9	3	-59	74	1	4	4	104	91	1	12	4	-51	1	8	7	5	174	-173
10	2	3	641	631	7	9	3	142	128	2	4	4	1115	1155	2	12	4	358	-352	9	7	5	492	-487
11	2	3	124	-137	8	9	3	96	84	3	4	4	82	86	3	12	4	-48	4	10	7	5	308	-321
12	2	3	173	178	9	9	3	129	113	4	4	4	560	558	4	12	4	282	-278	11	7	5	324	-321
13	2	3	-55	11	10	9	3	96	89	5	4	4	63	47	5	12	4	-44	-5	12	7	5	149	-165
14	2	3	70	74	11	9	3	210	188	6	4	4	435	444	6	12	4	255	-251	13	7	5	227	-226
15	2	3	-48	27	12	9	3	93	103	7	4	4	257	-230	1	1	5	238	214	0	8	5	647	-636
16	2	3	60	57	0	10	3	555	542	8	4	4	255	262	2	1	5	349	368	1	8	5	162	-134
17	2	3	-67	32	1	10	3	-62	11	9	4	4	242	-238	3	1	5	107	-113	2	8	5	606	-614
1	3	3	1190	1258	2	10	3	400	400	10	4	4	86	59	4	1	5	1050	990	3	8	5	166	-165
2	3	3	473	-470	3	10	3	78	80	11	4	4	-57	-42	5	1	5	440	-410	4	8	5	616	-629
3	3	3	457	534	4	10	3	359	345	12	4	4	151	130	6	1	5	657	637	5	8	5	146	-143
4	3	3	681	-674	5	10	3	85	79	13	4	4	-52	23	7	1	5	550	-546	6	8	5	463	-450
5	3	3	778	764	6	10	3	63	-6	14	4	4	84	77	8	1	5	421	406	7	8	5	176	-167
6	3	3	444	-424	7	10	3	231	223	15	4	4	-67	27	9	1	5	396	-372	8	8	5	385	-378
7	3	3	391	413	8	10	3	-51	-5	16	4	4	83	55	10	1	5	190	-177	9	8	5	119	-139
8	3	3	331	-337	9	10	3	167	163	1	5	4	189	194	11	1	5	157	-148	10	8	5	247	-237
9	3	3	634	641	10	10	3	76	62	2	5	4	159	-153	12	1	5	189	-181	11	8	5	73	-50
10	3	3	122	-120	11	10	3	100	94	3	5	4	228	437	13	1	5	137	-129	12	8	5	200	-202
11	3	3	474	454	1	11	3	-59	66	4	5	4	228	-197	14	1	5	59	-59	1	9	5	323	-317
12	3	3	-56	14	2	11	3	-58	28	5	5	4	704	676	15	1	5	86	-87	2	9	5	65	-33
13	3	3	498	493	3	11	3	-56	3	6	5	4	179	-174	16	1	5	-49	27	3	9	5	299	-301
14	3	3	-51	-5	4	11	3	-56	18	7	5	4	519	512	0	2	5	241	-173	4	9	5	107	-113
15	3	3	362	367	5	11	3	-54	47	8	5	4	-58	-6	1	2	5	150	144	5	9	5	233	-227
16	3	3	-67	72	6	11	3	-51	-6	9	5	4	328	331	2	2	5	516	-533	6	9	5	104	-111
0	4	3	1800	1752	7	11	3	68	45	10	5	4	-58	-69	3	2	5	667	620	7	9	5	250	-252
1	4	3	249	-231	8	11	3	97	-99	11	5	4	67	-55	4	2	5	629	-612	8	9	5	128	128
2	4	3	1855	1913	9	11	3	-42	37	12	5	4	65	60	5	2	5	263	258	9	9	5	188	-186
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5	4	3	557	-536	2	12	3	234	-227	15	5	4	159	-105	8	2	5	493	-475	0	10	5	70	-37
6	4	3	678	662	3	12	3	52	-47	0	6	4	749	-731	9	2	5	171	-166	1	10	5	-65	-61
7	4	3	207	-187	4	12	3	121	-115	1	6	4	118	-112	10	2	5	393	-383	2	10	5	68	-44
8	4	3	426	405	5	12	3	100	-95	2	6	4	676	-664	11	2	5	92	-98	3	10	5	133	-146
9	4	3	79	-83	6	12	3	-44	10	3	6	4	-58	83	12	2	5	223	-220	4	10	5	170	-175
10	4	3	614	612	7	12	3	122	-130	4	6	4	365	-347	13	2	5	131	-129	5	10	5	88	85
11	4	3	122	55	1	13	3	201	-210	5	6	4	-58	-28	14	2	5	175	-174	6	10	5	130	-149
12	4	3	731	723	2	13	3	-43	-7	6	6	4	101	103	15	2	5	-46	-7	7	10	5	160	149
13	4	3	88	77	3	13	3	132	-133	7	6	4	64	68	16	2	5	109	-132	8	10	5	164	-159
14	4	3	586	592	0	0	4	1004	1013	8	6	4	117	98	1	3	5	857	-880	9	10	5	190	196
15	4	3	-61	29	1	0	4	569	-586	9	6	4	70	94	2	3	5	291	-295	1	11	5	82	-77
16	4	3	355	364	2	0	4	1074	1118	10	6	4	200	184	3	3	5	909	-915	2	11	5	-58	40
1	5	3	1233	1282	3	0	4	596	-544	11	6	4	161	167	4	3	5	814	-763	3	11	5	-57	-39
2	5	3	108	-55	4	0	4	1741	1765	12	6	4	-52	-29	5	3	5	1097	-1065	4	11	5	-55	-25
3	5	3	1128	1137	5	0	4	664	-664	13	6	4	-49	55	6	3	5	425	-405	5	11	5	69	-55
4	5	3	288	265	6	0	4	1569	1514	14	6	4	58	-41	7	3	5	608	-606	6	11	5	-50	10
5	5	3	537	517	7	0	4	88	-80	1	7	4	684	-679	8	3	5	244	-243	7	11	5	93	-76
6	5	3	265	268	8	0	4	1255	1231	2	7	4	76	68	9	3	5	427	-423	0	12	5	-52	62
7	5	3	787	757	9	0	4	59	55	3	7	4	470	-457	10	3	5	-58	24	1	12	5	-51	4
8	5	3	392	382	10	0	4	386	344	4	7	4	-59	15	11	3	5	218	-212	2	12	5	-50	30
9	5	3	556	544	11	0	4	342	328	5	7	4	332	-309	12	3	5	-55	37	3	12	5	79	78
10	5	3	200	211	12	0	4	273	280	6	7	4	69	68	13	3	5	272	-267	4	12	5	-46	-26
11	5	3	395	356	13	0	4	-55	30	7	7	4	295	-287	14	3	5	-48	18	0	0	6	1748	-1583
12	5	3	93	82	14	0	4	94	52	8	7	4	134	125	15	3	5	210	-211	1	0	6	-66	41
13	5	3	498	492	15	0	4	-49	-39	9	7	4	163	-166	0	4	5	1256	-1217	2	0	6	1111	-1046
14	5	3	-51	-62	16	0	4	132	113	10	7	4	74	85	1	4	5	252	-266	3	0	6	-64	-58
15	5	3	298	316	1	1	4	1496	1479	11	7	4	102	-100	2	4	5	1300	-1297	4	0	6	785	-759
0	6	3	525	534	2	1	4	263	-240	12	7	4	-49	-31	3	4	5	703	-691	5	0	6	-63	-16
1	6	3	236	227	3	1	4	1558	1514	13	7	4	-44	-3	4	4	5	701	-715	6	0	6	640	-632
2	6	3	766	799	4	1	4	431	-389	14	7	4	-63	-44	5	4	5	340	-325	7	0	6	139	-123
3	6	3	242	234	5	1	4	1189	1172	0	8	4	160	156	6	4	5	663	-666	8	0	6	759	-750
4	6	3	888	906	6	1	4	130	-137	1	8	4	-62	-10	7	4	5	221	-218	9	0	6	89	-81
5	6	3	346	327	7	1	4	1171	1151	2	8	4	187	150	8	4	5	535	-537	10	0	6	766	-760
6	6	3	1067	1058	8	1	4	166	171	3	8	4	143	-153	9	4	5	99	95	11	0	6	367	-357
7	6	3	271	246	9	1	4	974	955	4	8	4	436	-438	10	4	5	578	-571	12	0	6	660	-678
8	6	3	903	901	10	1	4	73	48	5	8	4	105	99	11	4	5	176	176	13	0	6	83	-81
9	6	3	134	133	11	1	4	749	728	6	8	4	517	-517	12	4	5	422	-417	14	0	6	566	-600
10	6	3	635	633	12	1	4	-56	17	7	8	4	-58	11	13	4	5	197	196	15	0	6	-46	45
11	6	3	-56	-45	13	1	4	344	339	8	8	4	487	-479	14	4	5	324	-323	1	1	6	1173	-1104
12	6	3	277	266	14	1	4	-52	10	9	8	4	94	-68	15	4	5	-70	25	2	1	6	-64	16
13	6	3	59	-53	15	1	4	281	285	10	8	4	362	-358	1	5	5	883	-896	3	1	6	1048	-1031
14	6	3	156	184	16	1	4	-43	-3	11	8	4	120	-120	2	5	5	88	67	4	1	6	177	162
15	6	3	-70	-22	0	2	4	1648	1608	12	8	4	159	-165	3	5	5	814	-809	5	1	6	706	-697
1	7	3	475	500	1	2	4	-57	-27	13	8	4	-41	-44	4	5	5	137	138	6	1	6	99	80
2	7	3	65	-25	2	2	4	844	757	1	9	4	156	-158	5	5	5	715	-728	7	1	6	604	-602
3	7	3	713	727	3	2	4	79	93	2	9	4	-61	-16	6	5	5	109	101	8	1	6	148	-139
4	7	3	101	-103	4	2	4	867	875	3	9	4	306	-301	7	5	5	675	-695	9	1	6	513	-509
5	7	3	1182	1166	5	2	4	573	555	4	9	4	72	-55	8	5	5	288	306	10	1	6	89	-80
6	7	3	87	-75	6	2	4	785	796	5	9	4	418	-415	9	5	5	623	-629	11	1	6	612	-601
7	7	3	1026	1022	7	2	4	163	142	6	9	4	60	-52	10	5	5	224	231	12	1	6	-60	-30
8	7	3	309	-299	8	2	4	768	745	7	9	4	563	-567	11	5	5	546	-540	13	1	6	434	-445
9	7	3	725	734	9	2	4	-56	7	8	9	4	-54	-40	12	5	5	53	69	14	1	6	-51	-3
10	7	3	175	-158	10	2	4	803	796	9	9	4	451	-458	13	5	5	494	-488	15	1	6	342	-361
11	7	3	490	479	11	2	4	120	-121	10	9	4	-48	-23	14	5	5	-52	23	0	2	6	1025	-986
12	7	3	88	-58	12	2	4	719	706	11	9	4	353	-359	0	6	5	733	-735	1	2	6	-66	47

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	2	6	556	-563	0	10	6	268	249	7	7	7	242	241	3	5	8	190	191	4	5	9	210	215
3	2	6	147	-142	1	10	6	65	80	8	7	7	60	-90	4	5	8	111	117	5	5	9	377	-370
4	2	6	1115	-1124	2	10	6	256	267	9	7	7	254	256	5	5	8	87	-95	6	5	9	172	189
5	2	6	123	-118	3	10	6	-60	62	10	7	7	67	-72	6	5	8	-65	-64	7	5	9	385	-379
6	2	6	822	-818	4	10	6	352	358	11	7	7	275	291	7	5	8	128	-118	8	5	9	-55	31
7	2	6	299	284	5	10	6	103	106	0	8	7	359	354	8	5	8	103	-100	9	5	9	383	-406
8	2	6	624	-625	6	10	6	351	352	1	8	7	-66	0	9	5	8	-58	-51	10	5	9	-47	-29
9	2	6	251	251	7	10	6	65	53	2	8	7	281	282	10	5	8	103	-111	0	6	9	362	-352
10	2	6	257	-254	8	10	6	288	298	3	8	7	105	-83	11	5	8	-50	50	1	6	9	-65	-31
11	2	6	214	223	1	11	6	195	197	4	8	7	334	334	12	5	8	85	-86	2	6	9	314	-318
12	2	6	254	-246	2	11	6	159	171	5	8	7	156	-160	0	6	8	252	-249	3	6	9	-62	1
13	2	6	-55	0	3	11	6	224	224	6	8	7	294	296	1	6	8	-69	77	4	6	9	332	-336
14	2	6	136	-125	4	11	6	201	211	7	8	7	130	-127	2	6	8	-67	-41	5	6	9	-59	31
15	2	6	-56	-32	5	11	6	284	306	8	8	7	328	330	3	6	8	197	198	6	6	9	406	-410
1	3	6	702	-718	6	11	6	117	107	9	8	7	80	-94	4	6	8	154	-165	7	6	9	-55	51
2	3	6	233	-253	0	12	6	326	314	10	8	7	293	297	5	6	8	262	281	8	6	9	429	-444
3	3	6	737	-752	1	12	6	-46	-15	1	9	7	201	203	6	6	8	-63	-78	9	6	9	88	85
4	3	6	117	-106	1	1	7	926	882	2	9	7	-61	-15	7	6	8	147	161	1	7	9	455	-464
5	3	6	846	-851	2	1	7	-69	46	3	9	7	304	308	8	6	8	105	-96	2	7	9	139	-149
6	3	6	88	-60	3	1	7	278	269	4	9	7	-59	22	9	6	8	-54	-46	3	7	9	491	-509
7	3	6	854	-842	4	1	7	234	210	5	9	7	418	432	10	6	8	-50	1	4	7	9	207	-206
8	3	6	120	92	5	1	7	-68	-6	6	9	7	-55	43	11	6	8	67	-71	5	7	9	450	-466
9	3	6	637	-636	6	1	7	446	432	7	9	7	356	367	1	7	8	346	-350	6	7	9	179	-193
10	3	6	179	178	7	1	7	339	-332	8	9	7	51	47	2	7	8	-65	46	7	7	9	322	-334
11	3	6	290	-282	8	1	7	285	278	9	9	7	233	251	3	7	8	274	-286	8	7	9	-47	-2
12	3	6	197	206	9	1	7	101	-98	0	10	7	133	125	4	7	8	84	69	0	8	9	467	-465
13	3	6	110	-56	10	1	7	156	160	1	10	7	-57	30	5	7	8	112	-130	1	8	9	-57	-37
14	3	6	-51	-3	11	1	7	-61	-22	2	10	7	133	135	6	7	8	86	83	2	8	9	387	-383
15	3	6	79	-77	12	1	7	-58	13	3	10	7	93	98	7	7	8	70	-91	3	8	9	96	-99
0	4	6	83	34	13	1	7	124	126	4	10	7	154	149	8	7	8	56	73	4	8	9	317	-320
1	4	6	-65	-9	14	1	7	-48	-23	5	10	7	61	80	9	7	8	-50	-13	5	8	9	70	-78
2	4	6	-65	6	0	2	7	1009	954	6	10	7	172	176	10	7	8	-46	55	6	8	9	206	-221
3	4	6	93	77	1	2	7	-70	1	7	10	7	116	123	0	8	8	367	-361	7	8	9	77	-89
4	4	6	210	-207	2	2	7	889	874	1	11	7	-50	26	1	8	8	66	-75	1	9	9	157	-165
5	4	6	233	-216	3	2	7	147	-150	2	11	7	92	-93	2	8	8	324	-341	2	9	9	114	116
6	4	6	419	-420	4	2	7	535	526	3	11	7	51	22	3	8	8	174	-177	3	9	9	144	-142
7	4	6	207	-217	5	2	7	-68	-30	4	11	7	-45	-42	4	8	8	161	-151	4	9	9	107	106
8	4	6	394	-403	6	2	7	235	235	0	0	8	1514	1391	5	8	8	175	-187	0	0	10	102	-35
9	4	6	94	-83	7	2	7	79	77	1	0	8	137	-121	6	8	8	87	-73	1	0	10	82	-57
10	4	6	370	-377	8	2	7	128	114	2	0	8	1254	1183	7	8	8	118	-119	2	0	10	208	-199
11	4	6	70	-92	9	2	7	197	180	3	0	8	-73	60	8	8	8	119	-111	3	0	10	256	-241
12	4	6	241	-251	10	2	7	211	208	4	0	8	1085	972	9	8	8	-45	55	4	0	10	365	-361
13	4	6	60	43	11	2	7	71	31	5	0	8	-71	61	1	9	8	249	-255	5	0	10	241	-243
14	4	6	206	-217	12	2	7	292	305	6	0	8	579	576	2	9	8	120	-126	6	0	10	486	-475
1	5	6	112	100	13	2	7	-52	-48	7	0	8	120	-130	3	9	8	211	-218	7	0	10	171	-153
2	5	6	125	131	14	2	7	261	278	8	0	8	327	325	4	9	8	176	-175	8	0	10	549	-536
3	5	6	-66	-54	1	3	7	384	383	9	0	8	322	-319	5	9	8	174	-183	9	0	10	112	-105
4	5	6	176	181	2	3	7	163	-161	10	0	8	349	357	6	9	8	110	-118	10	0	10	435	-451
5	5	6	-65	-45	3	3	7	500	502	11	0	8	243	-258	7	9	8	185	-200	11	0	10	-44	-16
6	5	6	97	65	4	3	7	238	-221	12	0	8	314	308	0	10	8	327	-312	1	1	10	489	-483
7	5	6	82	72	5	3	7	490	486	13	0	8	92	-85	1	10	8	-51	13	2	1	10	71	-62
8	5	6	198	-206	6	3	7	339	-342	1	1	8	778	749	2	10	8	257	-264	3	1	10	476	-469
9	5	6	-63	25	7	3	7	641	626	2	1	8	135	131	3	10	8	50	47	4	1	10	-69	-36
10	5	6	185	-184	8	3	7	231	-230	3	1	8	790	767	4	10	8	285	-305	5	1	10	489	-485
11	5	6	113	-127	9	3	7	599	583	4	1	8	90	42	1	1	9	117	78	6	1	10	-64	-47
12	5	6	124	-136	10	3	7	80	-105	5	1	8	682	683	2	1	9	105	-100	7	1	10	514	-515
13	5	6	189	-169	11	3	7	445	452	6	1	8	-70	-2	3	1	9	-73	-35	8	1	10	-57	7
14	5	6	-55	-4	12	3	7	-56	8	7	1	8	614	605	4	1	9	142	-160	9	1	10	388	-394
0	6	6	452	-445	13	3	7	327	341	8	1	8	-66	15	5	1	9	159	-170	10	1	10	96	31
1	6	6	322	318	14	3	7	-62	14	9	1	8	378	383	6	1	9	126	109	11	1	10	286	-309
2	6	6	446	-465	0	4	7	141	35	10	1	8	-62	-53	7	1	9	152	-149	0	2	10	956	-916
3	6	6	337	341	1	4	7	118	-112	11	1	8	360	359	8	1	9	220	219	1	2	10	122	125
4	6	6	-67	44	2	4	7	317	327	12	1	8	-54	-80	9	1	9	253	-256	2	2	10	728	-712
5	6	6	298	306	3	4	7	162	-160	13	1	8	270	282	10	1	9	262	271	3	2	10	295	293
6	6	6	85	95	4	4	7	443	439	0	2	8	252	216	11	1	9	228	-236	4	2	10	560	-551
7	6	6	88	78	5	4	7	142	-146	1	2	8	-72	32	12	1	9	126	149	5	2	10	221	213
8	6	6	171	185	6	4	7	839	840	2	2	8	269	257	0	2	9	247	-226	6	2	10	281	-278
9	6	6	92	-85	7	4	7	-66	-4	3	2	8	-70	1	1	2	9	154	-151	7	2	10	129	123
10	6	6	127	124	8	4	7	713	717	4	2	8	438	433	2	2	9	214	-212	8	2	10	215	-217
11	6	6	65	-89	9	4	7	-64	-73	5	2	8	95	97	3	2	9	252	-258	9	2	10	104	97
12	6	6	81	93	10	4	7	536	544	6	2	8	531	511	4	2	9	276	-214	10	2	10	189	-192
13	6	6	69	-42	11	4	7	73	-58	7	2	8	146	119	5	2	9	152	-138	1	3	10	494	-483
1	7	6	72	-18	12	4	7	219	212	8	2	8	697	684	6	2	9	268	-277	2	3	10	-68	21
2	7	6	99	-125	13	4	7	-48	11	9	2	8	171	176	7	2	9	-66	69	3	3	10	378	-371
3	7	6	136	113	1	5	7	821	831	10	2	8	475	477	8	2	9	292	-294	4	3	10	110	107
4	7	6	-66	-9	2	5	7	197	207	11	2	8	219	216	9	2	9	165	171	5	3	10	213	-218
5	7	6	144	154	3	5	7	785	787	12	2	8	352	374	10	2	9	224	-226	6	3	10	120	125
6	7	6	-65	83	4	5	7	284	280	13	2	8	61	54	11	2	9	133	149	7	3	10	97	-68
7	7	6	208	207	5	5	7	879	877	1	3	8	297	297	12	2	9	150	-159	8	3	10	-54	1
8	7	6	122	139	6	5	7	174	182	2	3	8	177	-179	1	3	9	761	-758	9	3	10	53	-43
9	7	6	140	162	7	5	7	628	626	3	3	8	399	394	2	3	9	80	-69	10	3	10	66	-55
10	7	6	94	92	8	5	7	76	32	4	3	8	212	-229	3	3	9	570	-578	0	4	10	69	7
11	7	6	205	217	9	5	7	492	507	5	3	8	545	545	4	3	9	76	-67	1	4	10	-66	-31
12	7	6	-47	43	10	5	7	-58	45	6	3	8	-67	-46	5	3	9	398	-385	2	4	10	114	-101
0	8	6	749	727	11	5	7	292	294	7	3	8	519	522	6	3	9	159	-163	3	4	10	189	-179
1	8	6	174	-184	12	5	7	-50	50	8	3	8	132	116	7	3	9	270	-279	4	4	10	146	-148
2	8	6	598	623	13	5	7	165	179	9	3	8	446	446	8	3	9	101	-97	5	4	10	170	-178
3	8	6	204	-208	0	6	7	1027	987	10	3	8	127	127	9	3	9	216	-225	6	4	10	178	-177
4	8	6	409	411	1	6	7	166	162	11	3	8	284	292	10	3	9	108	-112	7	4	10	100	-106
5	8	6	284	-298	2	6	7	952	956	12	3	8	75	83	11	3	9	144	-146	8	4	10	199	-191
6	8	6	243	256	3	6	7	218	212	13	3	8	183	192	12	3	9	55	-71	9	4	10	-48	-66
7	8	6	66	-70	4	6	7	751	771	0	4	8	693	668	0	4	9	559	-543	1	5	10	72	86
8	8	6	209	205	5	6	7	139	129	1	4	8	132	-132	1	4	9	-69	65	2	5	10	-64	-81
9	8	6	105	90	6	6	7	465	495	2	4	8	544	532	2	4	9	688	-699	3	5	10	-61	9
10	8	6	101	113	7	6	7	82	74	3	4	8	172	-166	3	4	9	153	157	4	5	10	119	-128
11	8	6	76	91	8	6	7	317	315	4	4	8	425	420	4	4	9	595	-605	5	5	10	160	-158
1	9	6	586	607	9	6	7	78	93	5	4	8	166	-156	5	4	9	122	118	6	5	10	144	-141
2	9	6	177	-187	10	6	7	269	271	6	4	8	262	245	6	4	9	518	-532	7	5	10	247	-265
3	9	6	525	529	11	6	7	-51	11	7	4	8	177	-176	7	4	9	-61	-42	8	5	10	62	-42
4	9	6	211	-220	12	6	7	313	323	8	4	8	107	118	8	4	9	247	-242	9	5	10	187	-206
5	9	6	360	362	1	7	7	579	579	9	4	8	96	-87	9	4	9	168	-165	0	6	10	175	167
6	9	6	108	-100	2	7	7	158	-143	10	4	8	140	132	10	4	9	209	-217	1	6	10	61	-44
7	9	6	240	239	3	7	7	453	454	11	4	8	-53	-24	11	4	9	143	-148	2	6	10	-59	65
8	9	6	-53	-15	4	7	7	77	-70	12	4	8	65	47	1	5	9	346	-356	3	6	10	-57	9
9	9	6	131	126	5	7	7	256	242	1	5	8	494	491	2	5	9	235	243	4	6	10	-56	-22
10	9	6	-45	38	6	7	7	67	-46	2	5	8	213	222	3	5	9	349	-346	5	6	10	-54	45

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)
6	6	10	53	-89	5	1	11	271	279	4	3	11	94	-98	5	5	11	270	281	3	1	12	325	327
7	6	10	124	124	6	1	11	-56	16	5	3	11	223	228	6	5	11	107	105	4	1	12	55	-53
1	7	10	-55	-2	7	1	11	256	271	6	3	11	-52	-44	0	6	11	364	356	5	1	12	292	297
2	7	10	88	97	8	1	11	86	86	7	3	11	175	188	1	6	11	-51	3	6	1	12	-45	-52
3	7	10	-53	63	9	1	11	106	118	8	3	11	-45	-39	2	6	11	337	335	0	2	12	207	194
4	7	10	143	147	0	2	11	96	69	0	4	11	486	473	3	6	11	-49	18	1	2	12	61	57
5	7	10	115	119	1	2	11	-65	-31	1	4	11	62	63	4	6	11	327	339	2	2	12	186	193
6	7	10	104	120	2	2	11	145	134	2	4	11	430	437	1	7	11	132	122	3	2	12	213	211
0	8	10	85	62	3	2	11	-61	-38	3	4	11	69	57	0	0	12	423	414	4	2	12	262	270
1	8	10	-50	54	4	2	11	251	253	4	4	11	224	228	1	0	12	71	-82	5	2	12	197	217
2	8	10	103	100	5	2	11	-57	-7	5	4	11	-52	-13	2	0	12	408	429	1	3	12	240	259
3	8	10	-47	36	6	2	11	297	306	6	4	11	199	196	3	0	12	248	-262	2	3	12	120	120
4	8	10	199	215	7	2	11	-51	34	7	4	11	62	-75	4	0	12	385	394	3	3	12	166	168
1	1	11	192	184	8	2	11	220	230	1	5	11	542	556	5	0	12	232	-249	4	3	12	139	154
2	1	11	-64	14	1	3	11	126	121	2	5	11	-55	38	6	0	12	320	347	0	4	12	199	192
3	1	11	274	267	2	3	11	101	-107	3	5	11	458	475	1	1	12	335	343	1	4	12	-49	-35
4	1	11	-61	10	3	3	11	149	143	4	5	11	74	91	2	1	12	-53	-21	2	4	12	197	211
																				3	4	12	118	-116

Table 5. Bond lengths (Å) and angles (°). Standard deviations in parentheses. A prime denotes an atom generated by the twofold axis of symmetry.

Te − S(2) = 2.380	(2)	∠S(?) − Te − S(2′) = 97.61	(6)
S(1) − S(2) = 2.080	(2)	∠Te − S(2) − S(1) = 103.46	(7)
S(1) − O(1) = 1.433	(5)	∠S(2) − S(1) − O(1) = 103.1	(3)
S(1) − O(2) = 1.434	(5)	∠S(2) − S(1) − O(2) = 110.0	(3)
S(1) − C(1) = 1.779	(6)	∠S(2) − S(1) − C(1) = 104.5	(3)
C(1) − C(2) = 1.401	(9)	∠O(1) − S(1) − O(2) = 119.9	(3)
C(2) − C(3) = 1.379	(11)	∠O(1) − S(1) − C(1) = 109.6	(3)
C(3) − C(4) = 1.388	(11)	∠O(2) − S(1) − C(1) = 108.6	(3)
C(4) − C(5) = 1.400	(10)	∠S(1) − C(1) − C(2) = 119.0	(5)
C(5) − C(6) = 1.378	(10)	∠S(1) − C(1) − C(6) = 117.2	(5)
C(6) − C(1) = 1.390	(9)	∠C(1) − C(2) − C(3) = 117.6	(6)
		∠C(2) − C(3) − C(4) = 120.0	(7)
		∠C(3) − C(4) − C(5) = 121.0	(8)
		∠C(4) − C(5) − C(6) = 120.5	(7)
		∠C(5) − C(6) − C(1) = 117.1	(6)
		∠C(6) − C(1) − C(2) = 123.8	(6)

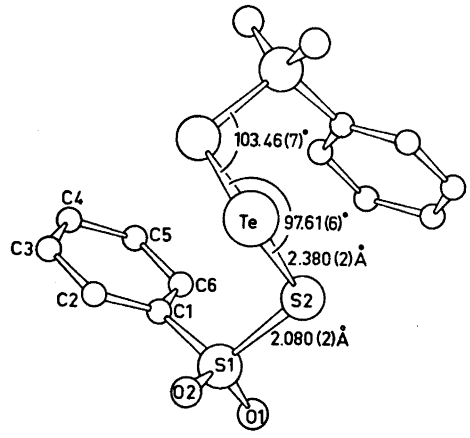

Fig. 1. The tellurium dibenzenethiosulphonate molecule as seen along the twofold rotation axis.

Since the molecule possesses a two-fold axis of symmetry, the non-planar S – S – Te – S – S chain occurs in the *trans* form, with the terminal sulphur atoms on opposite sides of the plane through the three middle atoms.

The greatest difference between the two investigations is in the Te – S(2) bond length, which is now found to be 2.380(2) Å. The older value is 2.41 Å, with a probable error of 0.03 – 0.04 Å. It is interesting to note that a Fourier map from the earlier study indicates a bond length of 2.39 Å.

The dihedral angle between the S(1) – S(2) – Te and the S(2) – Te – S(2′) planes is 97.4°. The S(1) – S(2) bond length, 2.080(2) Å, is in excellent agreement with the earlier value. In the complex, *trans*-dibenzenethiosulphonato-bis(trimethylenethiourea)tellurium(II),[1] the S – S bond length is 2.018(4) Å. This difference in the S – S bond lengths is thought to be caused by the difference in the nature of the Te – S bond in the complex relative to the uncomplexed compound, which is the subject of the present study.

The coordination around the S(1) atom is approximately tetrahedral, with the greatest deviation for the O(1) – S(1) – O(2) angle, which is 119.9(3)°. The bond lengths and angles involved in this coordination are in good agreement with the values given by Øyum and Foss.[2]

The atoms of a least squares plane through the benzene ring do not deviate more than 0.013 Å from the plane. S(1) is 0.095 Å from this plane, on the same side as Te, while O(1) and O(2) are 0.779 and 0.012 Å from it, on the opposite side.

Excluding the S(1) – S(2) bond length, the dimensions of the benzenethiosulphonate group are very similar to the dimensions found for the same group in the complex referred to above. In both compounds one of the oxygen atoms is near the plane of the benzene ring, and the C(6) – C(1) – C(2) angle is somewhat large.

Acknowledgement. I wish to thank Prof. Olav Foss for his interest. This investigation is in part financially supported by *Norges Almenvitenskapelige Forskningsråd (Norwegian Research Council for Science and Humanities)*.

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Received July 13, 1970.