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Refinement of the Crystal Structure of Monoclinic Barium Telluropentathionate Dihydrate

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The crystal structure of the monoclinic dimorph of barium telluropentathionate dihydrate, $BaTe(S_2O_3)_2.2H_2O$, has been refined by full-matrix least squares for 1306 non-zero reflections, collected by means of a single-crystal diffractometer, to a conventional R value of 0.031. The space group is $A2/m$ with four formula units per unit cell, and $a = 5.003(2)$ Å, $b = 10.588(3)$ Å, $c = 23.635(7)$ Å, $\beta = 98.61(5)°$.

Mirror plane symmetry is crystallographically required for the telluropentathionate ion, and the ion thus occurs in the *cis* form. The dimensions of the S–S–Te–S–S chain are: S(1)–S(2) = 2.104(3) Å, S(2)–Te = 2.377(3) Å, ∠S(1)–S(2)–Te = 103.79(9)°, ∠S(2)–Te–S(2′) = 100.29(8)°. The SSTe/STeS dihedral angles are 103.8°.

Barium telluropentathionate dihydrate, $BaTe(S_2O_3)_2.2H_2O$, occurs in a triclinic form when crystallized from aqueous methanol, and in a monoclinic form when crystallized from aqueous acetone, whereas a monoclinic trihydrate, $BaTe(S_2O_3)_2.3H_2O$, occurs on crystallization from water.[1]

The crystal structure of the monoclinic dihydrate was determined by Foss and Tjomsland,[2] using *0kl* and *h0l* Weissenberg photographs, taken with Cu$K\alpha$ radiation. No correction for absorption was made ($\mu = 531$ cm^{-1}), but reflections likely to be most heavily influenced by absorption were omitted from the refinement, which was carried out by difference electron density syntheses. Estimated standard deviations were 0.02 Å for S–S and S–T distances, and 1° for angles involving these atoms.

In connection with the crystal structure analyses of the trihydrate[3] and of barium selenopentathionate hydrates,[4,5] carried out with the aid of more modern facilities, it was thought of interest to refine the structure of the monoclinic dihydrate using the same methods.

EXPERIMENTAL

The crystals of monoclinic barium telluropentathionate dihydrate, $BaTe(S_2O_3)_2.2H_2O$, were obtained on addition of acetone to a solution of barium telluropentathionate in dilute perchloric acid.[1]

The crystal used for data collection was a prism along the a axis, bounded by (001), (011) and (0$\bar{1}$1), and terminated by (110) and (1$\bar{1}$0). The distance between the (001) boundary faces was 0.055 mm, and the maximum extensions along the a and b axes were 0.187 and 0.112 mm, respectively.

The intensity data were collected by means of a Siemens automatic single-crystal diffractometer using Mo$K\alpha$ radiation (Nb-filtered) and a scintillation counter.

The crystal was mounted with the a axis approximately parallel to the ϕ axis of the diffractometer, and the unit cell dimensions and setting angles for all reflections were calculated from the θ,χ and ϕ angles of 8 non-coplanar reciprocal vectors.

The procedure followed was similar to that described in a preceding paper.[3]

The net intensities of the two reference reflections, 080 and 008, decreased by about 5 % during the data collection.

Out of 1588 independent reflections attainable within $\theta = 28°$, 282 were found to have net intensities below three times its standard deviation. These reflections were assigned an intensity equal to this limit, and labelled as unobserved.

The net intensities were scaled by means of the reference reflections, and Lorentz and polarization corrections were applied. The linear absorption coefficient for Mo$K\alpha$ radiation is 63.5 cm^{-1}, and corrections were carried out using the Gaussian integration method.[6]

The scattering factor curves used for the structure factor calculations, and the computer programs used during the data collection, data processing, refinement, and drawing of illustrations are the same as in a preceding paper.[3]

The unit cell dimensions, calculated by means of a least squares program using the θ angles of 12 reflections ($\theta = 17 - 24°$) measured on the diffractometer, are $a = 5.003(2)$ Å, $b = 10.588(3)$ Å, $c = 23.635(7)$ Å, $\beta = 98.61(5)°$. The space group is $A2/m$ with four $BaTe(S_2O_3)_2.2H_2O$ formula units per unit cell.[1]

REFINEMENT

The first structure factor calculations were based on the atomic coordinates derived from the two-dimensional film data,[2] and an overall isotropic thermal factor, $\exp[-8\pi^2 U(\sin^2\theta/\lambda^2)]$ with $U = 0.040$ Å^2, was used. The resulting R value ($\sum||F_o| - |F_c||/\sum|F_o|$) was 0.154.

Refinement by least squares was then started, using a full-matrix least squares program minimizing the function

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

Here $W = 4(I_t - I_b)^2/F_o^2[I_t + I_b + k^2(I_t - I_b)^2]$, where I_t is the total intensity of a reflection, I_b is the background intensity, and k is the relative standard deviation in the scaling curve based on the reference reflections. Non-observed reflections for which $|F_c|$ is greater than the observable limit, are included in the refinement with $|F_o|$ equal to this limit.

With individual isotropic thermal parameters for all atoms the R value converged at 0.104. The values of U were in the region $0.027 - 0.047$ Å^2 for all atoms except the $H_2O(2)$ oxygen atom, for which U was 0.184 Å^2. On refinement of the occupancy factor for the $H_2O(2)$ oxygen atom this factor was reduced from 0.500 to 0.264. An attempt to use anisotropic refinement for this oxygen atom resulted in steadily increasing thermal parameters and decreasing x and z coordinates. The refinement converged at $U = 0.109$ Å^2 when an isotropic thermal parameter for $H_2O(2)$ was used.

With anisotropic thermal parameters for all atoms, except $H_2O(2)$, the final R value was 0.031. Unobserved reflections are included when $|F_c|$ is

greater than the observable limit. The maximum shift of a parameter in the last refinement cycle was about one tenth of its standard deviation.

A final difference electron density map showed no peaks higher than 0.7 e/Å^3. The water hydrogen atoms were not located.

Tables 1 and 2 give the final atomic parameters with standard deviations from least squares. The observed structure factors, and those calculated from the final parameters, are listed in Table 3.

Table 1. Atomic coordinates for barium telluropentathionate dihydrate. Origin at a centre of symmetry. Standard deviation are given in parentheses.

	x	y	z
Ba	0.19877(11)	0	0.20457(3)
S(1)	0.36898(32)	0.23827(15)	0.33787(7)
S(2)	0.40906(48)	0.17231(19)	0.42270(8)
Te	0.71446(14)	0	0.42420(3)
O(1)	0.6382(10)	0.2527(6)	0.3234(2)
O(2)	0.2325(11)	0.3578(5)	0.3437(2)
O(3)	0.2097(10)	0.1478(5)	0.3016(2)
$H_2O(1)$	0.7293(13)	0	0.2658(3)
$H_2O(2)$	0.1778(54)	0	0.0822(11)

Table 2. Thermal parameters expressed 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, and standard deviations are given in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Ba	255(3)	219(3)	391(3)	0	0	12(2)
S(1)	275(8)	208(7)	321(8)	−11(6)	43(6)	20(6)
S(2)	784(15)	316(9)	285(9)	109(10)	−3(8)	95(9)
Te	460(4)	327(4)	354(4)	0	0	−112(3)
O(1)	280(26)	598(37)	653(38)	−82(25)	182(31)	97(24)
O(2)	595(36)	244(26)	654(38)	166(38)	−3(25)	−69(29)
O(3)	366(26)	406(29)	395(27)	−61(23)	−105(23)	14(21)
$H_2O(1)$	234(33)	469(42)	299(36)	0	0	21(28)
$H_2O(2)$[a]	1087(79)					

[a] Occupancy factor for $H_2O(2)$ oxygen atom, 0.264 instead of 0.500, and the thermal parameter is expressed in the form exp $[-8\pi^2 U(\sin\theta/\lambda^2)]$.

RESULTS

Fig. 1. gives a view of the telluropentathionate ion as seen along the *a* axis, with principal bond lengths and angles. These values are nearly the same as those given by Foss and Tjomsland,[2] the greatest difference being that of 0.04 Å for the S–Te bond length. The ion possesses mirror plane symmetry, and the non-planar S–S–Te–S–S chain thus occurs in the *cis*

Table 3. Observed and calculated structure factors (× 10) for barium telluropentathionate dihydrate. A minus sign on $F(O)$ indicates an unobserved reflection.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
0	0	2	1746	-1752	0	6	16	271	305	1	0	2	1595	-1694	1	3	13	276	311	1	7-25	454	457
0	0	4	818	-1076	0	6	18	1343	-1274	1	0	4	737	692	1	3	15	653	712	1	7-23	-153	-53
0	0	6	480	-402	0	6	20	175	-215	1	0	6	1711	-1744	1	3	17	744	755	1	7-21	505	524
0	0	8	3725	-3637	0	6	22	658	-660	1	0	8	3218	3145	1	3	19	1015	976	1	7-19	774	-753
0	0	10	1178	1057	0	6	24	506	451	1	0	10	960	917	1	3	21	925	-933	1	7-17	353	-339
0	0	12	1234	1237	0	6	26	390	393	1	0	12	1520	-1534	1	3	23	313	-322	1	7-15	210	196
0	0	14	2156	2166	0	7	1	433	-462	1	0	14	535	557	1	3	25	603	-598	1	7-13	765	-778
0	0	16	353	-293	0	7	3	1338	-1360	1	0	16	1386	-1329	1	3	27	-157	111	1	7-11	1930	1946
0	0	18	573	-525	0	7	5	1090	1661	1	0	18	245	-251	1	3	29	534	520	1	7 -9	310	-312
0	0	20	-228	-387	0	7	7	487	488	1	0	20	727	728	1	4-23	-154	33		1	7 -7	178	-181
0	0	22	1392	-1405	0	7	9	538	509	1	0	22	502	460	1	4-20	-159	97		1	7 -5	-130	-62
0	0	24	1656	1659	0	7	11	433	-428	1	0	24	245	183	1	4-24	595	-609		1	7 -3	1610	-1646
0	0	26	645	638	0	7	13	591	-592	1	0	26	415	-405	1	4-22	325	-303		1	7 -1	757	749
0	0	28	-186	51	0	7	15	-139	-43	1	0	28	-164	83	1	4-20	181	192		1	7 1	-129	-114
0	0	30	199	234	0	7	17	537	-505	1	1	-31	-156	-69	1	4-18	334	-309		1	7 3	1577	1552
0	1	1	1314	-1535	0	7	19	1207	1199	1	1	-29	240	-221	1	4-16	1862	1907		1	7 5	164	143
0	1	3	1551	-1769	0	7	21	-172	55	1	1	-27	-151	-136	1	4-14	320	-312		1	7 7	1117	-1115
0	1	5	3219	3104	0	7	23	-152	-72	1	1	-25	876	862	1	4-12	942	-917		1	7 9	615	606
0	1	7	1056	924	0	7	25	-152	18	1	1	-23	-154	-207	1	4-10	144	-117		1	7 11	1820	-1822
0	1	9	1286	1250	0	8	0	2514	2514	1	1	-21	573	628	1	4 -8	2117	-2092		1	7 13	811	827
0	1	11	779	-839	0	8	2	577	-1004	1	1	-19	975	-913	1	4 -6	1613	1541		1	7 15	669	683
0	1	13	1575	-2013	0	8	4	-132	-22	1	1	-17	825	-790	1	4 -4	236	263		1	7 17	-166	-92
0	1	15	-135	145	0	8	6	203	200	1	1	-15	268	266	1	4 -2	2398	2434		1	7 19	559	566
0	1	17	771	-715	0	8	8	1226	-1231	1	1	-13	572	-641	1	4 0	479	-408		1	7 21	732	-719
0	1	19	1613	1643	0	8	10	541	533	1	1	-11	2782	2826	1	4 2	2476	-2397		1	7 23	-153	-72
0	1	21	408	404	0	8	12	-138	-35	1	1	-9	750	-730	1	4 4	757	795		1	7 25	271	-269
0	1	23	-148	-102	0	8	14	740	755	1	1	-7	-134	-15	1	4 6	2652	-2630		1	8-24	280	-290
0	1	25	172	-156	0	8	16	174	-217	1	1	-5	648	-676	1	4 8	2181	2187		1	8-22	380	-380
0	1	27	818	-818	0	8	18	216	-183	1	1	-3	2719	-2847	1	4 10	1422	1399		1	8-20	-151	67
0	1	29	182	168	0	8	20	-155	150	1	1	-1	2112	1942	1	4 12	642	-592		1	8-18	249	-215
0	2	0	2035	1725	0	8	22	734	-729	1	1	1	200	0	1	4 14	749	784		1	8-16	1257	1287
0	2	2	-122	-147	0	8	24	441	442	1	1	3	2226	2151	1	4 16	1725	-1674		1	8-14	175	-162
0	2	4	1591	1412	0	9	1	202	-189	1	1	5	709	660	1	4 18	393	-361		1	8-12	451	-472
0	2	6	2548	-2553	0	9	3	501	-520	1	1	7	2059	-2150	1	4 20	223	244		1	8-10	-140	111
0	2	8	1124	-1153	0	9	5	1078	1067	1	1	9	626	532	1	4 22	411	384		1	8 -8	1665	-1701
0	2	10	1646	1788	0	9	7	437	-645	1	1	11	2245	-2302	1	4 24	671	665		1	8 -6	939	922
0	2	12	1211	-1227	0	9	9	828	824	1	1	13	1232	1268	1	4 26	266	-271		1	8 -4	577	541
0	2	14	1666	1679	0	9	11	153	-133	1	1	15	754	814	1	4 28	-162	84		1	8 -2	994	957
0	2	16	231	359	0	9	13	867	-850	1	1	17	156	147	1	5-29	263	-199		1	8 0	-133	71
0	2	18	1143	-1074	0	9	15	646	630	1	1	19	686	693	1	5-27	-157	-78		1	8 2	1047	-1075
0	2	20	-142	117	0	9	17	653	-710	1	1	21	1199	-1224	1	5-25	834	841		1	8 4	-133	-11
0	2	22	532	-541	0	9	19	449	433	1	1	23	-149	25	1	5-23	256	-299		1	8 6	1595	-1603
0	2	24	336	287	0	9	21	182	123	1	1	25	256	-307	1	5-21	458	468		1	8 8	1426	1413
0	2	26	-151	66	0	9	23	-157	-48	1	1	27	220	191	1	5-19	659	-634		1	8 10	860	847
0	2	28	296	257	0	10	0	910	910	1	1	29	533	512	1	5-17	903	-887		1	8 12	334	-312
0	2	30	-137	100	0	10	2	-139	-114	1	2	-30	467	485	1	5-15	381	414		1	8 14	660	638
0	3	1	-125	-44	0	10	4	433	437	1	2	-28	202	-228	1	5-13	319	-283		1	8 16	1098	-1096
0	3	3	1499	-1558	0	10	6	514	-909	1	2	-26	541	533	1	5-11	1828	1843		1	8 18	402	-394
0	3	5	2415	2444	0	10	8	667	-687	1	2	-24	603	-620	1	5 -9	396	-359		1	8 20	-154	109
0	3	7	1300	-1214	0	10	10	849	857	1	2	-22	516	-899	1	5 -7	-124	33		1	9 22	363	337
0	3	9	1139	1152	0	10	12	480	-475	1	2	-20	560	615	1	5 -5	803	-759		1	8 24	425	434
0	3	11	189	89	0	10	14	751	765	1	2	-18	244	-195	1	5 -3	2090	-2086		1	9-23	-162	-131
0	3	13	1573	-1557	0	10	16	203	197	1	2	-16	1422	1450	1	5 -1	1778	1786		1	9-21	610	597
0	3	15	829	861	0	10	18	522	-523	1	2	-14	222	195	1	5 1	-125	-149		1	9-19	-154	-59
0	3	17	1011	-977	0	10	20	-159	-26	1	2	-12	521	-543	1	5 3	1239	1269		1	9-17	678	-684
0	3	19	672	627	0	11	1	351	-361	1	2	-10	946	-923	1	5 5	828	873		1	9-15	156	-134
0	3	21	-146	31	0	11	3	636	-829	1	2	-8	2682	-2631	1	5 7	1781	-1755		1	9-13	532	-519
0	3	23	-144	-76	0	11	5	1342	1334	1	2	-6	2823	2856	1	5 9	-127	35		1	9-11	1071	1046
0	3	25	332	263	0	11	7	-147	115	1	2	-4	538	-550	1	5 11	1323	-1293		1	9 -9	147	152
0	3	27	563	-552	0	11	9	380	429	1	2	-2	2432	2350	1	5 13	966	969		1	9 -7	632	606
0	3	29	201	201	0	11	11	-176	-206	1	2	0	1732	1649	1	5 15	397	446		1	9 -5	212	-239
0	4	0	3698	3736	0	11	13	650	-840	1	2	2	3973	-3994	1	5 17	291	288		1	9 -3	1732	-1708
0	4	2	1046	-1054	0	11	15	-152	45	1	2	4	-130	-48	1	5 19	470	468		1	9 -1	614	604
0	4	4	-151	66	0	11	17	287	-282	1	2	6	1251	-1226	1	5 21	1137	-1114		1	9 1	458	-466
0	4	6	498	-446	0	11	19	794	772	1	2	8	1243	1192	1	5 23	-155	126		1	9 3	947	927
0	4	8	1667	-1682	0	12	0	1545	1540	1	2	10	1268	1247	1	5 25	213	-213		1	9 5	1174	1159
0	4	10	739	793	0	12	2	290	-283	1	2	12	863	843	1	5 27	-162	131		1	9 7	744	-743
0	4	12	277	217	0	12	4	-147	-44	1	2	14	-138	-60	1	6-28	393	-435		1	9 -141		-114
0	4	14	1665	1119	0	12	6	318	-346	1	2	16	2089	-2133	1	6-26	-151	137		1	9 11	1140	-1129
0	4	16	252	-219	0	12	8	876	-876	1	2	18	363	351	1	6-24	429	-434		1	9 13	262	225
0	4	18	303	-245	0	12	10	433	437	1	2	20	343	-338	1	6-22	462	-459		1	9 15	223	230
0	4	20	155	-132	0	12	12	-170	118	1	2	22	318	280	1	6-20	935	959		1	9 17	483	500
0	4	22	928	-916	0	12	14	666	708	1	2	24	1289	1278	1	6-18	-143	52		1	9 19	621	611
0	4	24	763	704	0	13	1	266	-269	1	2	26	538	-519	1	6-16	621	641		1	9 21	740	-716
0	4	26	-153	121	0	13	3	255	-252	1	2	28	-156	-71	1	6-14	457	-431		1	10-20	397	425
0	4	28	-155	13	0	13	5	643	648	1	3	-29	254	-274	1	6-12	778	-769		1	10-18	209	-180
0	5	1	1278	-1280	0	13	7	-157	-173	1	3	-27	478	-477	1	6-10	274	-262		1	10-16	714	696
0	5	3	1046	-1039	0	13	9	517	518	1	3	-25	536	531	1	6 -8	1071	-1091		1	10-14	-149	-40
0	5	5	2470	2481	0	13	11	157	-202	1	3	-23	-148	39	1	6 -6	2402	2397		1	10-12	276	-298
0	5	7	178	190	0	14	0	555	601	1	3	-21	1015	1110	1	6 -4	189	-173		1	10-10	365	-352
0	5	9	1372	1379	1	0	-30	566	576	1	3	-19	421	-397	1	6 -2	330	332		1	10 -8	846	-840
0	5	11	646	-633	1	0	-28	263	-272	1	3	-17	1135	-1089	1	6 0	349	330		1	10 -6	1220	1206
0	5	13	1596	-2016	1	0	-26	302	-315	1	3	-15	-136	-54	1	6 2	2353	-2373		1	10 -4	371	-375
0	5	15	369	379	1	0	-24	459	-457	1	3	-13	1349	-1338	1	6 4	388	403		1	10 -2	888	901
0	5	17	637	-580	1	0	-22	213	-166	1	3	-11	1908	1975	1	6 6	-126	-55		1	10 0	378	353
0	5	19	1238	1254	1	0	-20	698	748	1	3	-9	568	995	1	6 8	1057	1074		1	10 2	1569	-1604
0	5	21	505	452	1	0	-18	-139	107	1	3	-7	580	599	1	6 10	459	469		1	10 4	393	378
0	5	23	-152	-129	1	0	-16	1461	1443	1	3	-5	566	-527	1	6 12	-138	-104		1	10 6	553	563
0	5	25	247	-237	1	0	-14	580	-590	1	3	-3	2776	-2812	1	6 14	284	-237		1	10 8	552	560
0	5	27	692	-693	1	0	-12	1659	-1672	1	3	-1	-129	166	1	6 16	1257	-1232		1	10 10	529	530
0	6	4	419	404	1	0	-10	704	679	1	3	1	491	-494	1	6 18	601	609		1	10 12	-149	138
0	6	6	2052	-2048	1	0	-8	1882	-1891	1	3	3	1990	2077	1	6 20	-146	63		1	10 14	-148	52
0	6	8	1720	-1750	1	0	-6	2601	2486	1	3	5	2122	2137	1	6 22	186	147		1	10 16	996	-996
0	6	10	1489	1484	1	0	-4	1837	1662	1	3	7	1316	-1301	1	6 24	628	632		1	10 18	305	310
0	6	12	298	-316	1	0	-2	357	161	1	3	9	201	-190	1	6 26	576	-565		1	10 20	-159	-123
0	6	14	1817	1851	1	0	0	1404	-1565	1	3	11	2289	-2295	1	7-27	-160	-95		1	11-19	543	-527

Table 3. 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)
1	11	-17	315	-295	2	2	-28	429	-463	2	5	11	363	-338	2	9	19	496	-479	3	1	9	-145	-69
1	11	-15	391	354	2	2	-26	206	-210	2	5	13	1635	1679	2	10	-20	-158	-4	3	1	11	1144	1174
1	11	-13	455	-432	2	2	-24	-158	-154	2	5	15	517	-509	2	10	-18	-153	33	3	1	13	684	-662
1	11	-11	595	582	2	2	-22	952	988	2	5	17	-148	131	2	10	-16	-158	163	3	1	15	1170	-1164
1	11	-9	-145	-24	2	2	-20	-147	84	2	5	19	561	-543	2	10	-14	1056	-1054	3	1	17	165	145
1	11	-7	353	-353	2	2	-18	203	205	2	5	21	385	-412	2	10	-12	521	522	3	1	19	439	-441
1	11	-5	179	-147	2	2	-16	157	142	2	5	23	309	290	2	10	-10	-145	-87	3	1	21	940	954
1	11	-3	724	-722	2	2	-14	2448	-2492	2	5	25	-156	11	2	10	-8	258	219	3	1	23	322	285
1	11	-1	574	564	2	2	-12	917	924	2	6	-26	-152	-54	2	10	-6	897	911	3	1	25	-158	-57
1	11	1	-139	-66	2	2	-10	-131	14	2	6	-24	280	-271	2	10	-4	743	-731	3	2	-28	324	336
1	11	3	782	777	2	2	-8	807	804	2	6	-22	437	459	2	10	-2	-142	51	3	2	-26	178	209
1	11	5	-146	58	2	2	-6	2309	2317	2	6	-20	346	-317	2	10	0	894	-897	3	2	-24	238	-230
1	11	7	757	-771	2	2	-4	1753	-1698	2	6	-18	350	346	2	10	2	390	387	3	2	-22	440	467
1	11	9	287	288	2	2	-2	204	-223	2	6	-16	561	573	2	10	4	381	372	3	2	-20	522	-515
1	11	11	794	-790	2	2	0	2587	-2594	2	6	-14	1455	-1479	2	10	6	-145	75	3	2	-18	181	-192
1	11	13	433	499	2	2	2	769	785	2	6	-12	722	714	2	10	8	889	913	3	2	-16	290	-322
1	11	15	391	371	2	2	4	1097	1131	2	6	-10	518	-515	2	10	10	923	-914	3	2	-14	479	464
1	11	17	-156	-69	2	2	6	534	517	2	6	-8	148	134	2	10	12	208	-212	3	2	-12	607	581
1	12	-14	368	-386	2	2	8	1955	1956	2	6	-6	1181	1197	2	10	14	-150	-55	3	2	-10	469	-533
1	12	-12	383	-387	2	2	10	1994	-1960	2	6	-4	517	-518	2	10	16	245	-222	3	2	-8	1558	1571
1	12	-10	172	179	2	2	12	899	-851	2	6	-2	605	607	2	10	18	598	573	3	2	-6	2199	-2203
1	12	-8	545	-551	2	2	14	257	-216	2	6	0	1900	-1899	2	11	-17	172	159	3	2	-4	-128	-113
1	12	-6	730	767	2	2	16	320	-267	2	6	2	622	584	2	11	-15	-159	-96	3	2	-2	742	732
1	12	-4	-144	55	2	2	18	1248	1242	2	6	4	-135	93	2	11	-13	358	367	3	2	0	1030	-980
1	12	-2	259	227	2	2	20	338	313	2	6	6	176	-157	2	11	-11	156	122	3	2	2	2113	2112
1	12	0	224	-194	2	2	22	261	227	2	6	8	1784	1795	2	11	-9	615	-609	3	2	4	274	-280
1	12	2	726	-717	2	2	24	771	-758	2	6	10	1113	-1089	2	11	-7	573	553	3	2	6	260	-253
1	12	4	444	412	2	2	26	326	-330	2	6	12	191	-177	2	11	-5	832	-832	3	2	8	750	-718
1	12	6	525	-527	2	3	-29	-161	-5	2	6	14	284	-283	2	11	-3	222	219	3	2	10	693	-677
1	12	8	745	750	2	3	-27	734	712	2	6	16	490	-475	2	11	-1	732	732	3	2	12	617	647
1	12	10	293	280	2	3	-25	-156	76	2	6	18	657	669	2	11	1	434	-441	3	2	14	244	-224
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1	12	14	227	239	2	3	-21	293	-248	2	6	22	485	486	2	11	5	638	-615	3	2	18	-155	-143
1	13	-11	621	606	2	3	-19	1067	-1033	2	6	24	427	-425	2	11	7	-150	-103	3	2	20	676	-694
1	13	-9	238	-241	2	3	-17	1021	1042	2	7	-25	-160	133	2	11	9	-154	-127	3	2	22	157	175
1	13	-7	228	260	2	3	-15	434	419	2	7	-23	339	-369	2	11	11	-149	27	3	2	24	579	-573
1	13	-5	-154	-55	2	3	-13	1302	1296	2	7	-21	251	234	2	11	13	650	671	3	3	-27	185	172
1	13	-3	906	-878	2	3	-11	-133	-6	2	7	-19	811	-903	2	11	15	418	-396	3	3	-25	550	-546
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1	13	1	331	-266	2	3	-7	227	247	2	7	-15	147	155	2	12	-12	291	193	3	3	-21	-155	-88
1	13	3	367	373	2	3	-5	1567	-1505	2	7	-13	420	412	2	12	-10	411	-394	3	3	-19	-148	88
1	13	5	423	408	2	3	-3	1619	1615	2	7	-11	483	463	2	12	-8	347	328	3	3	-17	823	819
1	13	7	538	-484	2	3	-1	2473	2434	2	7	-9	1088	-1109	2	12	-6	460	449	3	3	-15	1079	-1112
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2	0	-30	-162	-155	2	3	3	344	422	2	7	-5	1106	-1104	2	12	-2	542	551	3	3	-11	661	-685
2	0	-28	-154	-130	2	3	5	2830	-2810	2	7	-3	582	575	2	12	0	738	-751	3	3	-9	272	-263
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2	0	-24	784	-780	2	3	9	144	166	2	7	1	637	-645	2	12	4	260	259	3	3	-5	255	-253
2	0	-22	466	467	2	3	11	613	648	2	7	3	986	968	2	12	6	-151	-29	3	3	-3	1213	1207
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2	0	-2	2162	2061	2	4	-26	318	329	2	7	23	474	469	3	0	-28	-159	-4	3	3	17	239	230
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2	0	2	526	376	2	4	-22	700	711	2	8	-22	625	628	3	0	-24	476	487	3	3	21	677	669
2	0	4	729	730	2	4	-20	745	737	2	8	-20	466	454	3	0	-22	704	721	3	3	23	-159	86
2	0	6	-135	-30	2	4	-18	451	-447	2	8	-18	205	-227	3	0	-20	1007	-1029	3	4	-28	-160	99
2	0	8	1691	1674	2	4	-16	-139	1	2	8	-16	-145	-41	3	0	-18	320	288	3	4	-26	-154	-48
2	0	10	582	-581	2	4	-14	1276	-1250	2	8	-14	567	-1002	3	0	-16	993	-979	3	4	-24	246	299
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2	0	14	1269	-1248	2	4	-10	318	-326	2	8	-10	147	-170	3	0	-12	2254	2217	3	4	-20	847	-857
2	0	16	271	-263	2	4	-8	1405	1402	2	8	-8	1206	1191	3	0	-10	479	-453	3	4	-18	565	577
2	0	18	1352	1315	2	4	-6	1598	1615	2	8	-6	764	776	3	0	-8	1210	1151	3	4	-16	563	-595
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2	0	24	267	-268	2	4	0	2060	-2027	2	8	0	1748	-1792	3	0	-2	248	-292	3	4	-10	592	-597
2	0	26	651	-654	2	4	2	199	-122	2	8	2	244	208	3	0	0	-152	115	3	4	-8	440	456
2	0	28	-165	123	2	4	4	1404	1416	2	8	4	1128	1112	3	0	2	2388	2373	3	4	-6	707	-719
2	1	-25	186	-215	2	4	6	695	684	2	8	6	253	244	3	0	4	383	-364	3	4	-4	290	-259
2	1	-27	446	426	2	4	8	968	956	2	8	8	773	788	3	0	6	522	459	3	4	-2	558	-567
2	1	-25	-151	76	2	4	10	1380	-1331	2	8	10	588	-578	3	0	8	1091	-1062	3	4	0	275	264
2	1	-23	522	-251	2	4	12	335	-252	2	8	12	237	-238	3	0	10	1924	-1902	3	4	2	1839	1879
2	1	-21	254	264	2	4	14	876	-822	2	8	14	669	-664	3	0	12	1093	1099	3	4	4	1169	-1153
2	1	-19	1258	-1245	2	4	16	-145	-55	2	8	16	-154	73	3	0	14	194	188	3	4	6	637	662
2	1	-17	885	905	2	4	18	1469	1440	2	8	18	1094	1100	3	0	16	935	919	3	4	8	614	-592
2	1	-15	-140	36	2	4	20	237	-244	2	8	20	264	-280	3	0	18	392	390	3	4	10	1369	-1326
2	1	-13	427	410	2	4	22	386	341	2	8	22	343	324	3	0	20	611	-643	3	4	12	939	962
2	1	-11	990	991	2	4	24	341	-352	2	9	-23	156	-219	3	0	22	442	-425	3	4	14	-145	-5
2	1	-9	1651	-1702	2	4	26	591	-622	2	9	-21	-160	-141	3	0	24	583	-591	3	4	16	654	676
2	1	-7	-132	60	2	5	-27	309	258	2	9	-19	756	-787	3	1	-29	-158	0	3	4	18	-150	19
2	1	-5	1142	-1208	2	5	-25	-153	-2	2	9	-17	859	868	3	1	-27	280	275	3	4	20	345	-341
2	1	-3	743	714	2	5	-23	-156	-116	2	9	-15	-151	172	3	1	-25	690	-705	3	4	22	-165	-167
2	1	-1	1803	1760	2	5	-21	-152	192	2	9	-13	366	338	3	1	-23	-151	40	3	4	24	496	-545
2	1	1	515	-561	2	5	-19	587	-978	2	9	-11	459	441	3	1	-21	208	-212	3	5	-27	313	317
2	1	3	1470	1488	2	5	-17	829	818	2	9	-9	1337	-1361	3	1	-19	324	326	3	5	-25	623	-625
2	1	5	2233	-2241	2	5	-15	221	-209	2	9	-7	-142	-96	3	1	-17	982	973	3	5	-23	-154	-2
2	1	7	653	-630	2	5	-13	345	359	2	9	-5	472	-454	3	1	-15	433	-459	3	5	-21	182	-183
2	1	9	232	269	2	5	-11	854	855	2	9	-3	776	763	3	1	-13	351	332	3	5	-19	172	214
2	1	11	-139	-107	2	5	-9	1429	-1423	2	9	-1	912	918	3	1	-11	1593	-1627	3	5	-17	871	870
2	1	13	1758	1767	2	5	-7	269	270	2	9	1	150	-114	3	1	-9	229	-235	3	5	-15	358	-373
2	1	15	454	-464	2	5	-5	917	-914	2	9	3	385	378	3	1	-7	937	903	3	5	-13	395	362
2	1	17	192	185	2	5	-3	422	437	2	9	5	1700	-1709	3	1	-5	-133	8	3	5	-11	1334	-1341
2	1	19	825	-833	2	5	-1	1089	1100	2	9	7	-145	76	3	1	-3	2120	2085	3	5	-9	280	-308
2	1	21	473	-450	2	5	1	224	-158	2	9	9	213	227	3	1	-1	1012	-1013	3	5	-7	767	768
2	1	23	471	439	2	5	3	1089	1109	2	9	11	-144	68	3	1	1	858	-823	3	5	-5	156	-129
2	1	25	-154	38	2	5	5	1958	-1945	2	9	13	1258	1260	3	1	3	719	-726	3	5	-3	1853	1842
2	1	27	568	555	2	5	7	-131	5	2	9	15	525	-525	3	1	5	958	-951	3	5	-1	801	-793
2	2	-30	-156	-8	2	5	9	-133	-7	2	9	17	202	-180	3	1	7	1869	1874	3	5	1	782	-750

Table 3. 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)
3	5	3	431	-465	3	10	-8	750	737	4	2	20	-159	-13	4	7	7	394	286	5	2	0	-147	-16
3	5	5	587	-564	3	10	-6	895	-895	4	3	-25	291	-282	4	7	9	596	-599	5	2	2	710	-705
3	5	7	1440	1443	3	10	-4	-145	-78	4	3	-23	342	346	4	7	11	-153	127	5	2	4	-158	184
3	5	9	-143	142	3	10	-2	179	184	4	3	-21	175	-166	4	7	13	719	-750	5	2	6	246	215
3	5	11	855	889	3	10	0	434	-450	4	3	-19	538	549	4	7	15	383	428	5	2	8	313	321
3	5	13	540	-538	3	10	2	1016	1023	4	3	-17	214	-243	4	8	-18	174	128	5	2	10	705	700
3	5	15	523	-515	3	10	4	180	-200	4	3	-15	255	-274	4	8	-16	215	-203	5	2	12	866	-915
3	5	17	-152	90	3	10	6	-148	45	4	3	-13	-149	-125	4	8	-14	651	651	5	2	14	-165	34
3	5	19	438	-441	3	10	8	333	-238	4	3	-11	591	-590	4	8	-12	339	-345	5	2	16	178	-232
3	5	21	776	839	3	10	10	411	-422	4	3	-9	1135	1125	4	8	-10	-147	31	5	3	-21	229	-180
3	5	23	311	258	3	10	12	337	338	4	3	-7	-140	-77	4	8	-8	-163	111	5	3	-19	-161	73
3	6	-26	-169	160	3	10	14	181	-184	4	3	-5	301	281	4	8	-6	961	-984	5	3	-17	697	-695
3	6	-24	-156	-18	3	11	-15	418	-421	4	3	-3	177	95	4	8	-4	862	859	5	3	-15	570	557
3	6	-22	674	709	3	11	-13	277	244	4	3	-1	1481	-1454	4	8	-2	-146	-83	5	3	-13	-154	95
3	6	-20	421	-436	3	11	-11	391	-411	4	3	1	463	487	4	8	0	286	273	5	3	-11	192	233
3	6	-18	355	-368	3	11	-9	390	-423	4	3	3	320	-311	4	8	2	244	252	5	3	-9	397	408
3	6	-16	565	-557	3	11	-7	345	349	4	3	5	579	967	4	8	4	584	-612	5	3	-7	962	-957
3	6	-14	173	142	3	11	-5	-155	137	4	3	7	404	391	4	8	6	-149	-48	5	3	-5	209	174
3	6	-12	776	749	3	11	-3	712	716	4	3	9	418	-403	4	8	8	435	-446	5	3	-3	646	-668
3	6	-10	-139	-66	3	11	-1	488	-482	4	3	11	-149	57	4	8	10	614	615	5	3	-1	537	536
3	6	-8	1527	1518	3	11	1	-149	-11	4	3	13	1190	-1183	4	8	12	-163	153	5	3	1	740	741
3	6	-6	1778	-1781	3	11	3	349	-356	4	3	15	499	503	4	8	14	-157	-9	5	3	3	368	-375
3	6	-4	713	-712	3	11	5	652	-644	4	3	17	206	207	4	9	-17	179	-172	5	3	5	614	635
3	6	-2	517	488	3	11	7	579	954	4	3	19	242	254	4	9	-15	314	-313	5	3	7	1168	-1169
3	6	0	809	-839	3	11	9	-159	-91	4	4	-24	-162	124	4	9	-13	-156	136	5	3	9	-157	-77
3	6	2	1857	1917	3	12	-8	381	368	4	4	-22	-155	-96	4	9	-11	394	-405	5	3	11	199	156
3	6	4	312	276	3	12	-6	484	-439	4	4	-20	354	-388	4	9	-9	520	525	5	3	13	-165	66
3	6	6	262	-240	3	12	-4	252	-279	4	4	-18	211	235	4	9	-7	-148	54	5	3	15	616	727
3	6	8	872	-752	3	12	-2	164	-121	4	4	-16	191	-197	4	9	-5	232	233	5	4	-20	539	537
3	6	10	675	-669	3	12	0	-159	-69	4	4	-14	1023	1021	4	9	-3	-149	-122	5	4	-18	-160	30
3	6	12	485	483	3	12	2	896	915	4	4	-12	611	-630	4	9	-1	764	-759	5	4	-16	218	-219
3	6	14	-150	-81	3	12	4	281	-295	4	4	-10	-141	-11	4	9	1	484	467	5	4	-14	324	322
3	6	16	1141	1153	4	0	-26	297	-271	4	4	-8	211	206	4	9	3	465	-458	5	4	-12	802	-777
3	6	18	-158	90	4	0	-24	172	-145	4	4	-6	1679	-1668	4	9	5	623	635	5	4	-10	245	227
3	6	20	658	-703	4	0	-22	167	-159	4	4	-4	1524	1519	4	9	7	403	397	5	4	-8	-148	-76
3	6	22	-161	-3	4	0	-20	274	-230	4	4	-2	-139	87	4	9	9	463	-486	5	4	-6	553	552
3	7	-23	-152	144	4	0	-18	404	400	4	4	0	-141	109	4	9	11	-160	136	5	4	-4	253	239
3	7	-21	221	-225	4	0	-16	-171	45	4	4	2	476	487	4	10	-12	-154	-47	5	4	-2	458	-454
3	7	-19	242	225	4	0	-14	1016	1016	4	4	4	574	-939	4	10	-10	273	-272	5	4	0	335	350
3	7	-17	757	757	4	0	-12	902	-923	4	4	6	307	-290	4	10	-8	-151	-33	5	4	2	1113	-1104
3	7	-15	518	-511	4	0	-10	281	-319	4	4	8	486	-466	4	10	-6	472	-469	5	4	4	264	259
3	7	-13	242	221	4	0	-8	-156	104	4	4	10	538	959	4	10	-4	592	599	5	4	6	606	621
3	7	-11	866	-854	4	0	-6	1249	-1248	4	4	12	255	249	4	10	-2	186	-190	5	4	8	-166	-179
3	7	-9	223	-225	4	0	-4	1773	1479	4	4	14	-150	-83	4	10	0	518	535	5	4	10	556	601
3	7	-7	611	551	4	0	-2	185	159	4	4	16	448	442	4	10	2	-151	21	5	4	12	361	-404
3	7	-5	-140	103	4	0	0	211	220	4	4	18	579	-1101	4	10	4	750	-787	5	4	14	212	-312
3	7	-3	1228	1183	4	0	2	-156	-130	4	4	20	-167	59	4	10	6	322	349	5	5	-19	-162	89
3	7	-1	805	-819	4	0	4	1029	-1114	4	5	-23	455	491	4	10	8	463	-462	5	5	-17	377	-379
3	7	1	239	-249	4	0	6	173	-177	4	5	-21	255	-239	4	11	-7	247	-238	5	5	-15	422	437
3	7	3	563	-582	4	0	8	328	-307	4	5	-19	444	459	4	11	-5	-152	-79	5	5	-13	-158	-168
3	7	5	623	-622	4	0	10	1211	1229	4	5	-17	503	-512	4	11	-3	171	84	5	5	-11	166	177
3	7	7	1442	1447	4	0	12	169	163	4	5	-15	455	-473	4	11	-1	715	-714	5	5	-9	380	349
3	7	9	244	-259	4	0	14	165	-123	4	5	-13	296	279	4	11	1	356	375	5	5	-7	805	-803
3	7	11	711	722	4	0	16	225	209	4	5	-11	325	-320	5	0	-22	471	-466	5	5	-5	376	395
3	7	13	327	-321	4	0	18	1163	-1169	4	5	-9	1072	1080	5	0	-20	513	545	5	5	-3	482	-462
3	7	15	868	-863	4	0	20	251	264	4	5	-7	-140	-66	5	0	-18	-162	-113	5	5	-1	203	205
3	7	17	-154	115	4	1	-27	-168	-149	4	5	-5	-140	-71	5	0	-16	252	-262	5	5	1	570	569
3	7	19	249	-213	4	1	-25	-159	-77	4	5	-3	254	-270	5	0	-14	543	492	5	5	3	286	-297
3	8	-22	234	188	4	1	-23	563	567	4	5	-1	1168	-1161	5	0	-12	779	-761	5	5	5	426	429
3	8	-20	670	-675	4	1	-21	288	-290	4	5	1	877	873	5	0	-10	432	439	5	5	7	792	-792
3	8	-18	408	414	4	1	-19	541	551	4	5	3	308	-299	5	0	-8	362	-369	5	5	9	-156	89
3	8	-16	331	-326	4	1	-17	595	-608	4	5	5	557	968	5	0	-6	543	539	5	5	11	-157	-44
3	8	-14	-149	-123	4	1	-15	504	-503	4	5	7	299	321	5	0	-4	323	303	5	5	13	-159	36
3	8	-12	1050	1036	4	1	-13	353	351	4	5	9	878	-886	5	0	-2	666	-700	5	6	-18	-156	-51
3	8	-10	568	-636	4	1	-11	475	-504	4	5	11	-152	142	5	0	0	754	743	5	6	-16	-159	33
3	8	-8	432	425	4	1	-9	1352	1336	4	5	13	758	-830	5	0	2	1176	-1139	5	6	-14	237	152
3	8	-6	561	-566	4	1	-7	-142	-61	4	5	15	380	447	5	0	4	-162	133	5	6	-12	144	-135
3	8	-4	259	-253	4	1	-5	146	-146	4	5	17	318	342	5	0	6	665	679	5	6	-10	595	621
3	8	-2	-140	60	4	1	-3	170	-172	4	5	19	194	208	5	0	8	364	-343	5	6	-8	456	-452
3	8	0	-140	3	4	1	-1	1514	-1487	4	6	-22	380	-413	5	0	10	738	705	5	6	-6	204	208
3	8	2	1139	1120	4	1	1	1054	1053	4	6	-20	171	-170	5	0	12	435	-428	5	6	-4	591	579
3	8	4	658	-702	4	1	3	335	-349	4	6	-18	653	663	5	0	14	-172	-227	5	6	-2	599	-612
3	8	6	269	306	4	1	5	1076	1051	4	6	-16	320	-335	5	0	16	238	-231	5	6	0	291	266
3	8	8	315	-350	4	1	7	496	493	4	6	-14	657	669	5	1	-23	-159	-32	5	6	2	481	-498
3	8	10	829	-841	4	1	9	1356	-1346	4	6	-12	155	-179	5	1	-21	174	-145	5	6	4	-151	103
3	8	12	760	782	4	1	11	-154	146	4	6	-10	622	-621	5	1	-19	200	138	5	6	6	-157	125
3	8	14	-151	-79	4	1	13	569	-965	4	6	-8	144	-135	5	1	-17	487	-485	5	6	8	-156	96
3	8	16	404	376	4	1	15	499	501	4	6	-6	251	-258	5	1	-15	417	416	5	6	10	548	571
3	8	18	-155	54	4	1	17	417	394	4	6	-4	848	866	5	1	-13	-158	-69	5	7	-15	268	262
3	9	-21	-159	68	4	1	19	204	220	4	6	-2	242	-247	5	1	-11	220	211	5	7	-13	-157	77
3	9	-19	-155	28	4	1	21	255	280	4	6	0	910	923	5	1	-9	367	352	5	7	-11	-150	77
3	9	-17	271	288	4	2	-26	-165	-132	4	6	2	438	-452	5	1	-7	826	-832	5	7	-9	245	226
3	9	-15	418	-444	4	2	-24	372	363	4	6	4	1162	-1174	5	1	-5	335	347	5	7	-7	457	-461
3	9	-13	364	332	4	2	-22	419	-358	4	6	6	676	665	5	1	-3	601	-590	5	7	-5	174	179
3	9	-11	655	-668	4	2	-20	404	-385	4	6	8	606	-596	5	1	-1	306	302	5	7	-3	524	-518
3	9	-9	-152	128	4	2	-18	639	644	4	6	10	556	590	5	1	1	678	676	5	7	-1	334	341
3	9	-7	611	615	4	2	-16	643	-662	4	6	12	663	686	5	1	3	309	-303	5	7	1	399	433
3	9	-5	523	-529	4	2	-14	945	897	4	6	14	268	-324	5	1	5	503	493	5	7	3	290	-280
3	9	-3	551	944	4	2	-12	-147	99	4	6	16	-158	-82	5	1	7	959	-938	5	7	5	468	474
3	9	-1	600	-604	4	2	-10	523	-495	4	6	18	515	-545	5	1	9	-163	55	5	7	7	641	-695
3	9	1	257	-243	4	2	-8	188	-170	4	7	-21	190	-192	5	1	11	-160	-72	5	7	9	-158	-40
3	9	3	-146	128	4	2	-6	728	-722	4	7	-19	319	329	5	1	13	-163	106	5	8	-12	598	-621
3	9	5	450	-455	4	2	-4	542	938	4	7	-17	410	-396	5	1	15	639	700	5	8	-10	238	208
3	9	7	791	766	4	2	-2	464	-476	4	7	-15	185	-220	5	2	-22	214	-186	5	8	-8	227	-152
3	9	9	158	-200	4	2	0	1373	1358	4	7	-13	193	213	5	2	-20	284	291	5	8	-6	414	419
3	9	11	420	436	4	2	2	-144	-100	4	7	-11	465	-476	5	2	-18	-166	103	5	8	-4	269	296
3	9	13	342	-326	4	2	4	1493	-1462	4	7	-9	1048	1034	5	2	-16	-161	99	5	8	-2	413	-436
3	9	15	436	-446	4	2	6	709	703	4	7	-7	201	-188	5	2	-14	-158	41	5	8	0	323	319
3	9	17	238	257	4	2	8	1001	-988	4	7	-5	-146	-124	5	2	-12	953	-954	5	8	2	770	-800
3	10	-18	-155	-7	4	2	10	563	549	4	7	-3	146	106	5	2	-10	650	637	5	8	4	-159	119
3	10	-16	264	-251	4	2	12	589	993	4	7	-1	1067	-1081	5	2	-8	367	-374	5	8	6	446	462
3	10	-14	181	120	4	2	14	236	-279	4	7	1	689	671	5	2	-6	397	409	5	9	-7	725	-743
3	10	-12	342	326	4	2	16	-156	57	4	7	3	197	-177	5	2	-4	825	808	5	9	-5	236	253
3	10	-10	216	-223	4	2	18	686	-706	4	7	5	618	619	5	2	-2	710	-670	5	9	-3	297	-321

Table 3. Continued.

H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(C)	F(C)	H	K	L	F(O)	F(C)	H	K	L	F(O)	F(C)
6	9	-1	-163	150	6	1	-17	-165	47	6	2	-14	-164	-55	6	3	-11	267	234	6	4	-6	597	602
6	0	-16	209	201	6	1	-15	424	385	6	2	-12	-168	-91	6	3	-9	344	-353	6	4	-4	483	-494
6	0	-14	495	-511	6	1	-13	327	-328	6	2	-10	375	372	6	3	-7	-155	-77	6	4	-2	222	-216
6	0	-12	-170	129	6	1	-11	309	276	6	2	-8	-159	-119	6	3	-5	445	449	6	4	0	200	205
6	0	-10	566	523	6	1	-9	458	-459	6	2	-6	314	315	6	3	-3	279	-263	6	4	2	180	-136
6	0	-8	445	-443	6	1	-7	-161	-161	6	2	-4	646	-662	6	3	-1	545	556	6	4	4	480	527
6	0	-6	627	612	6	1	-5	386	364	6	2	-2	-155	130	6	3	1	403	-419	6	5	-11	173	168
6	0	-4	417	-412	6	1	-3	249	-236	6	2	0	-157	41	6	3	3	-161	-101	6	5	-9	367	-356
6	0	-2	441	-372	6	1	-1	771	762	6	2	2	342	-300	6	3	5	-162	-197	6	5	-7	-159	-41
6	0	0	-174	100	6	1	1	494	-526	6	2	4	853	858	6	3	7	222	-203	6	5	-5	230	225
6	0	2	-177	-46	6	1	3	-165	-22	6	2	6	256	-284	6	4	-12	203	211	6	5	-3	185	-199
6	0	4	575	553	6	1	5	-170	-148	6	2	8	-166	-74	6	4	-10	414	413	6	5	-1	679	688
6	0	6	-177	-157	6	1	7	384	-384	6	3	-15	303	292	6	4	-8	519	-509	6	5	1	458	-462
6	0	8	262	260	6	2	-16	185	189	6	3	-13	369	-383										

form, the two terminal sulphur atoms being located on the same side of the plane through the three middle atoms.

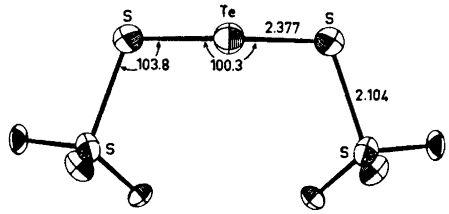

Fig. 1. The *cis* form of the telluropentathionate ion in $BaTe(S_2O_3)_2.2H_2O$ as seen along the *a* axis. The ellipsoids represent 50 % probability; the thermal parameters of the oxygen atoms are halved in size relative to those of the tellurium and sulphur atoms.

Table 4. Dimensions of the *cis* form of the telluro- and selenopentathionate ions in the dihydrates of the barium salts. For the left column X = Te, and for the right column X = Se. A prime denotes an atom generated by the mirror plane through X. Standard deviations are given in parentheses.

	$BaTe(S_2O_3)_2.2H_2O$	$BaSe(S_2O_3)_2.2H_2O$[4]
S(1) – S(2)	2.104(3) Å	2.096(3) Å
S(2) – X	2.377(3)	2.180(3)
∠S(1) – S(2) – X	103.79(9)°	103.96(11)°
∠S(2) – X – S(2′)	100.29(8)°	103.06(11)°
∠S(1)S(2)X/S(2)XS(2′)	103.8°	106.0°
S(1) – O(1)	1.446(6) Å	1.432(5) Å
S(1) – O(2)	1.447(5)	1.441(6)
S(1) – O(3)	1.443(5)	1.444(6)
∠S(2) – S(1) – O(1)	107.5(2)°	107.1(3)°
∠S(2) – S(1) – O(2)	100.3(3)°	99.1(3)°
∠S(2) – S(1) – O(3)	108.1(2)°	108.6(3)°
∠O(1) – S(1) – O(2)	113.0(3)°	113.1(3)°
∠O(1) – S(1) – O(3)	112.4(3)°	112.9(3)°
∠O(2) – S(1) – O(3)	114.5(3)°	114.7(3)°
∠XS(2)S(1)/S(2)S(1)O(1)	49.1°	50.6°
∠XS(2)S(1)/S(2)S(1)O(2)	167.3°	168.3°
∠XS(2)S(1)/S(2)S(1)O(3)	72.5°	71.7°
∠S(2)S(1)O(1)/S(2)S(1)O(2)	118.2°	117.7°
∠S(2)S(1)O(1)/S(2)S(1)O(3)	121.6°	122.3°
∠S(2)S(1)O(2)/S(2)S(1)O(3)	120.2°	120.0°

The dimensions of the ion calculated from the atomic coordinates of Table 1 are listed in the left column of Table 4. The standard deviations given include estimated uncertainties in unit cell dimensions. The non-bonded distances in the S(1) – S(2) – Te – S(2′) – S(1′) chain are: S(1) – Te = 3.530(2) Å, S(1) – (2′) = 4.779(3) Å, S(2) – S(2′) = 3.631(3) Å, S(1) – S(1′) = 5.003(3) Å.

Crystal structure determinations of barium selenopentathionate dihydrate [4,7] and trihydrate [5] have shown that the dimensions of the selenopentathionate ions are very nearly the same in these two hydrates. The ions have the same rotational isomeric form as the telluropentathionate ion in the present salt. For comparison, the dimensions of the selenopentathionate ion in the dihydrate of its barium salt are listed in the right column of Table 4.

The only significant difference in the dimensions of the two ions, except the S – X bond length and the S – X – S bond angle, is in the dihedral angles between the S – S – X and S – X – S planes. Variations in the degrees of rotation about S – X bonds are also found in the two halves of a S – S – X – S – S chain when symmetry is not crystallographically required.[3,8–10]

The difference between the S – Te and S – Se bond lengths is in accordance with the difference in the single covalent bond radii for tellurium, 1.37 Å, and selenium, 1.17 Å.[11] The bond lengths are in both cases 0.03 Å shorter than the sum of the single covalent bond radii for the atoms involved.

The S – Te – S bond angle is about 3° smaller than the S – Se – S bond angle which is again about 3° smaller than the middle S – S – S bond angle in barium pentathionates.[12] The same tendency of decreasing bond angles for the middle atom is found in the isomorphous series of penta-, selenopenta-, and telluropentathionate ions in the *trans* form.[8–10]

The bond lengths and angles of the sulphonate groups are in good agreement with those of the selenopentathionate ion and, as seen from the dihedral angles listed, there is no difference in the orientation of the S – O bonds relative to the X – S – S planes.

Each barium ion is surrounded by nine oxygen atoms, with Ba – O distances ranging from 2.747 Å to 2.938 Å. Three of the oxygen atoms coordinated to

Table 5. Distances (Å), and angles (°) between directions, from barium ion to oxygen atoms. Standard deviations of the distances and angles are 0.005 – 0.007 Å and 0.1 – 0.2°, respectively, except for the distance and angles involving $H_2O(2)$, which have standard deviations of 0.026 Å and 0.7°.

			I	II	III	IV	V	VI	VII	VIII
I	$H_2O(1)$	(x,y,z)	2.827							
II	$H_2O(1)$	$((x-1,y,z)$	2.938	120.4						
III	$H_2O(2)$	(x,y,z)	2.878	113.9	125.7					
IV	O(3)	(x,y,z)	2.770	71.1	60.7	145.6				
V	O(3)	$(x,\bar{y},z)$	2.770	71.1	60.7	145.6	68.8			
VI	O(1)	$(1-x,\frac{1}{2}-y,\frac{1}{2}-z)$	2.849	80.8	113.1	74.7	72.7	137.8		
VII	O(1)	$(1-x,-\frac{1}{2}+y,\frac{1}{2}-z)$	2.849	80.8	113.1	74.7	137.8	72.7	133.5	
VIII	O(2)	$(\bar{x},\frac{1}{2}-y,\frac{1}{2}-z)$	2.747	146.7	64.2	71.0	87.2	124.8	68.6	130.0
IX	O(2)	$(\bar{x},-\frac{1}{2}+y,\frac{1}{2}-z)$	2.747	146.7	64.2	71.0	124.8	87.2	130.0	68.6 66.5

one barium ion are from water molecules and the remaining six are from five different telluropentathionate ions. The distances and angles of the Ba – O coordination are listed in Table 5. Again there are only small deviations between these values and those given for the barium selenopentathionate hydrates.[4,5]

Fig. 2, which is a stereoscopic view as seen normal to the *a* crystal plane, shows the formation of layers through Ba – O coordination. The layers are

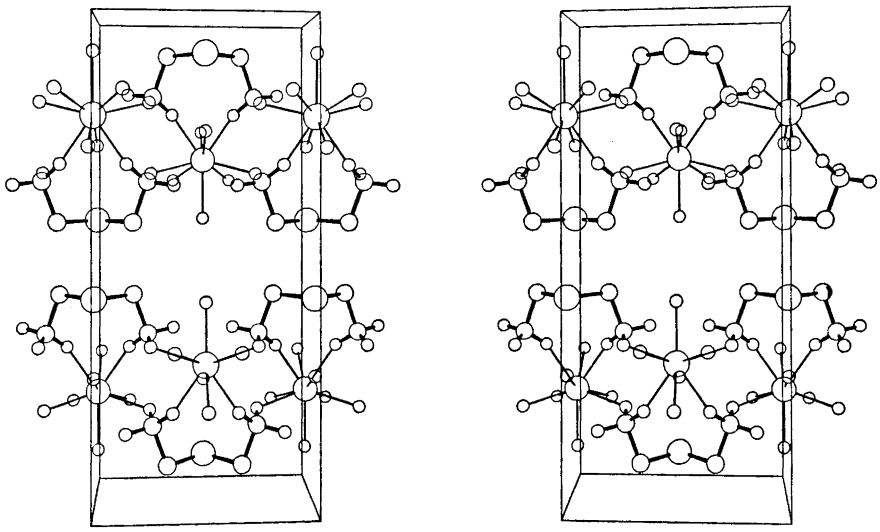

Fig. 2. A stereoscopic view of the cell packing in $BaTe(S_2O_3)_2.2H_2O$ as seen normal to the *a* crystal plane. The Ba – O coordination is indicated by the thin lines.

parallel to the *c* crystal plane and are the reason for the pronounced tendency of cleavage along this plane.[2] The relation between neighbour layers is through twofold rotation axes in the interfaces. Similar layers in barium selenopentathionate dihydrate and trihydrate are related through glide planes, and twofold screw axes, respectively.

The water molecule $H_2O(1)$ is coordinated to two barium ions and probably forms hydrogen bonds to O(1) and the image of O(1) across the mirror plane in which the water oxygen atom is located. The O – O distances are 3.067(7) Å, and the O – O – O angle is 121.5(2)°. The water molecule $H_2O(2)$ is coordinated to one barium ion, and does not seem to form any hydrogen bonds. The reason for the low occupancy factors and high termal parameters in the least squares refinement for the $H_2O(2)$ oxygen atoms in the present crystals and in the crystals of $BaSe(S_2O_3)_2.2H_2O$ [4] is probably that these water molecules are not so firmly held in the lattice. This is also shown by the fact that these water molecules are replaced by organic molecules when solvates are formed.[13]

Table 6 give the principal dimensions of the telluropentathionate ions as found in the crystals of $BaTe(S_2O_3)_2.2H_2O$ and $BaTe(S_2O_3)_2.3H_2O$.[3] The main

Table 6. Dimensions of the S – S – Te – S – S chain in the crystals of barium telluropentathionate dihydrate (*cis*) and trihydrate (*trans*). Standard deviations are given in parentheses.

	$BaTe(S_2O_3)_2.2H_2O$	$BaTe(S_2O_3)_2.3H_2O$[3]
S – S	2.104(3) Å	2.102(2) Å, 2.090(2) Å
S – Te	2.377(3) Å	2.384(2) Å, 2.392(2) Å
∠S – S – Te	103.79(9)°	103.08(5)°, 102.59(5)°
∠S – Te – S	100.29(8)°	96.02(4)°
∠SSTe/STeS	103.8°	87.7°, 98.7°

difference is the rotational isomerism: The rotations of the S – S bonds 87.7° and 98.7° to different sides of the plane through the three middle atoms in the trihydrate, and the rotations of the S – S bonds 103.8° to the same side of the plane in the dihydrate. The S – Te – S bond angles differ by 4.27°, whereas the remaining dimensions do not differ more from one ion to the other than they differ within the ion of the trihydrate, where symmetry is not crystallographically required. The larger dihedral angles and S – Te – S bond angle in the *cis* form might be due to space requirements of the sulphonate groups.

The closest interionic Te – Te, Te – S, and S – S approaches are 4.235(1) Å, 3.930(2) Å, and 3.631(3) Å, respectively.

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