The Crystal Structure of Rubidium Telluropentathionate

Hemitrihydrate

KJARTAN MARØY

Chemical Institute, University of Bergen, Bergen, Norway

The salt, $Rb_2Te(S_2O_3)_2.1\frac{1}{2}H_2O$, crystallizes in the orthorhombic space group *Pbcn* (No. 60) with eight formula units per cell, and $a = 21.320(10)$ Å, $b = 9.446(4)$ Å, $c = 12.437(5)$ Å. The structure has been determined by X-ray methods, and refined by least squares for the $hk0 - hk7$, $h0l$, and $h1l$ reflections.

The telluropentathionate ion occurs in the *trans* form in this salt, the sulphonate groups being located on opposite sides of the plane through the three middle atoms. The dimensions of the $S - S - Te - S - S$ chain are: $S(1) - S(2) = 2.116(11)$ Å, $S(2) - Te = 2.364(9)$ Å, $Te - S(4) = 2.370(7)$ Å, $S(4) - S(5) = 2.126(9)$ Å, $\angle S(1) - S(2) - Te = 103.3(4)°$, $\angle S(2) - Te - S(4) = 100.1(3)°$, $\angle Te - S(4) - S(5) = 102.8(3)°$. The dihedral angles, $S(1)S(2)Te/S(2)TeS(4) = 77.7°$ and $S(2)TeS(4)/TeS(4)S(5) = 89.2°$.

The dihedral angle of about 90° between the planes of the valencies of adjacent divalent sulphur atoms gives rise to two rotational-isomeric forms of the pentathionate ion; a *trans* form with the terminal sulphur atoms on opposite sides of the plane through the three middle atoms, and a *cis* form with the terminal sulphur atoms on the same side of the plane.[1] The same holds for the selenopentathionate and telluropentathionate ions, where the middle atom of the five-membered chain is selenium or tellurium instead of sulphur.

The *cis* form occurs in three barium pentathionates,[2-4] and in barium selenopentathionate dihydrate,[5] barium selenopentathionate trihydrate,[6] and barium telluropentathionate dihydrate.[7] The *trans* form has so far been found in the crystals of ammonium telluropentathionate [8] and barium telluropentathionate trihydrate.[9]

The present structure determinations of three representatives, $Rb_2Te(S_2O_3)_2.1\frac{1}{2}H_2O$, $(NH_4)_2Se(S_2O_3)_2.1\frac{1}{2}H_2O$, and $K_2S(S_2O_3)_2.1\frac{1}{2}H_2O$, of a series of isomorphous alkali (and ammonium) telluropentathionate, seleno-pentathionate, and pentathionate hemitrihydrates,[10] show that in these salts, the *trans* form occurs. The crystal structure of rubidium telluropentathionate hemitrihydrate is described here. Some features of the structure have been briefly reported earlier.[11]

EXPERIMENTAL

Sodium telluropentathionate dihydrate, $Na_2Te(S_2O_3)_2.2H_2O$, was prepared from tellurium dioxide and sodium thiosulphate.[12] The rubidium salt, $Rb_2Te(S_2O_3)_2.1\frac{1}{2}H_2O$, crystallizes on addition of aqueous rubidium chloride to a solution of the sodium salt in dilute hydrochloric acid.[10]

The crystals are orange red, and occur as prisms elongated in the direction of the c axis. The unit cell dimensions were redetermined by means of a least squares program, using the diffraction angles of reflections measured from zero-layer Weissenberg photographs around the b and c axes. Cu$K\alpha$ radiation was used, and the Weissenberg camera radius was checked with sodium chloride, $a = 5.6394$ Å (Ref. 13, p. 122). The unit cell is orthorhombic, with axial lengths $a = 21.320(10)$ Å, $b = 9.446(4)$ Å, $c = 12.437(5)$ Å. The space group, from systematic absences ($0kl$: $k = 2n + 1$, $h0l$: $l = 2n + 1$, and $hk0$: $h + k = 2n + 1$), is $Pbcn$ (No. 60), and there are eight formula units per unit cell.[10]

Using the multiple-film technique, integrated zero-layer and equi-inclination Weissenberg photographs were taken with Cu$K\alpha$ radiation (Ni-filtered) for the $hk0 - hk7$, $h0l$ and $h1l$ reflections. The intensities were estimated visually. Altogether, 1186 independent reflections were observed with measurable intensities; the unobserved reflections were set equal to the observable limit. The effects of spot extension and contraction in the equi-inclination photographs were reduced by horizontal integration, and the reflections were estimated on the upper and lower parts of the films. In the region where α_1 and α_2 reflections are split, only α_1 reflections were estimated, and the resulting intensities were increased by $2 - 50$ %, according to an experimental scale.

Lorentz and polarization corrections were applied, and a weight factor, W_0 used in the least squares weighting scheme, was calculated for each of the reflections. This weight factor is based on the number of readings and the estimated reliability of the individual readings.

The intensities were eventually corrected for absorption ($\mu = 364$ cm^{-1}), by the method described by Coppens et al.[14] The number of grid points used along the a, b, and c axes, respectively, were 6, 6, and 12 for the crystal rotating about the c axis, and 8 along all three axes for the crystal rotating about the b axis. Crystal dimensions are shown in Table 1.

Table 1. Distances between pairs of faces of the crystals used for collecting intensity data.

Pairs of faces	Crystal rotating about the c axis	Crystal rotating about the b axis
(100) and ($\bar{1}$00)	0.0106 cm	0.0084 cm
(110) and ($\bar{1}\bar{1}$0)	0.0099	0.0072
(1$\bar{1}$0) and ($\bar{1}$10)	0.0089	0.0072
(001) and (00$\bar{1}$)	0.0196	0.0084

The calculated structure factors were based on the scattering curves given in *International Tables for X-ray Crystallography*,[13] Table 3.3.1B for rubidium ion and tellurium, and Table 3.3.1A for sulphur and oxygen. The scattering curves for rubidium ion and tellurium were corrected for anomalous dispersion, using the $\Delta f'$ and $\Delta f''$ values calculated by Cromer,[17] and taking the amplitude of f as the corrected value.

Most of the computer programs used were made available by the Chemical Department of X-ray Crystallography, Weizmann Institute of Science, Rehovoth, Israel, and modified for use on the IBM 360/50H computer by Dr. D. Rabinovich. The program used for secondary extinction correction is written by Mr. K. Maartmann-Moɔ, and the program used for Fourier summations by Mr. E. Alver, both of this Institute. All calculations were carried out on an IBM 360/50H computer.

THE STRUCTURE ANALYSIS

A three-dimensional Patterson synthesis was calculated, based on 1105 observed $hk0 - hk7$ reflections, brought to the same scale by comparison with $h1l$ reflections. The Te $-$ Te vectors $u = \frac{1}{2} - 2x$, $v = \frac{1}{2}$, $w = 0$, and $u = \frac{1}{2} - 2x$, $v = \frac{1}{2} - 2y$, $w = \frac{1}{2}$, appearing at $(0.358, \frac{1}{2}, 0)$ and $(0.358, \frac{1}{2}, \frac{1}{2})$, respectively, gave $x = 0.071$ and $y = 0.0$. Because of overlap, it was not possible to determine the z coordinate. From the Patterson map of the isomorphous salt, $(NH_4)_2Se$ $(S_2O_3)_2 \cdot 1\frac{1}{2}H_2O$,[15] the coordinates for selenium were found to be $x = 0.078$, $y = 0.0$, $z = 0.079$ or 0.171. These z coordinates for tellurium would give a too short Te $-$ Te distance, 3.6 Å. The first structure factor calculations were therefore based on tellurium with coordinates, $x = 0.071$, $y = 0.0$, $z = 0.088$. Fourier summations were made of 178 l even reflections, with signs based on the tellurium contributions. Tellurium does not contribute to l odd reflections, since $y = 0$. The resulting map, which had false symmetry due to the omission of l odd reflections, indicated two possible sets of rubidium coordinates. The correct set was found by trial. The tellurium and rubidium contributions gave the probable signs of 514 reflections, including 101 l odd reflections. In the resulting map, one of the sulphonate sulphur atoms had to be placed close to a twofold axis. This was assumed to indicate that the z coordinate chosen for tellurium was the wrong one, and it was changed to 0.150. The sulphur and oxygen atoms could now be placed on the basis of subsequent electron density maps.

Full-matrix least squares refinement, using isotropic temperature parameters for all atoms, brought the reliability index, $R = \sum ||F_o| - |F_c|| / \sum |F_o|$, down to 0.145. The function minimized in the least squares program was

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

with $W = [(Ka_1)^2 + (a_2F_o)^2/4W_o]^{-1}$. Here W_o is the weight factor, mentioned earlier, based on the reliability of the individual intensities, and the constants a_1 and a_2 were put equal to 2.0 and 0.5, respectively. Unobserved reflections were included with $|F_o|$ equal to the observable limit, when $|F_c|$ exceeded this limit.

The number of observed reflections was increased to 1186 by adding the $h0l$ and $h1l$ reflections, and absorption corrections were carried out. The observed structure factors were then corrected for secondary extinction by means of a computer program, using the method of Zachariasen.[16] The formula used is $F_{corr} = KF_o(1 + \beta C I_o)$, where $\beta = 2(1 + \cos^4 2\theta)/(1 + \cos^2 2\theta)^2$, and C was found to be 1.13×10^{-7}. The absorption term in β was omitted, since the intensities had been corrected for absorption.

Least squares refinement, with anisotropic temperature parameters for the rubidium, tellurium, and sulphur atoms, reduced the R index to the final value of 0.091, with non-observed reflections included when $|F_c|$ exceeds the observable limit. In the last two cycles, all of the 94 parameters, including the overall scale factor, were refined simultaneously. In the last refinement cycle, no calculated shift of a parameter exceeded half the standard deviation of that parameter. The correctness of the structure was checked by a three-dimensional difference electron density map, based on observed and calculated

structure factors from the last refinement cycle. The difference map showed no peaks higher than 1.2 e/Å³.

The final atomic parameters, with standard deviations from the least squares output, are listed in Tables 2 and 3. The observed structure factors, and the calculated ones based on the parameters of Tables 2 and 3, are listed in Table 4.

Table 2. Atomic coordinates for rubidium telluropentathionate hemitrihydrate, in fractions of cell edges. Origin at a centre of symmetry. Standard deviations from least squares are given in parentheses.

	x	y	z
Rb(1)	0.2610(1)	0.2519(3)	0.4697(2)
Rb(2)	0.3922(1)	0.0856(4)	0.1899(3)
S(1)	0.0535(3)	0.2801(8)	−0.0374(7)
S(2)	0.0862(3)	0.0687(9)	−0.0315(7)
Te	0.0715(1)	0.0048(2)	0.1505(2)
S(4)	0.1585(3)	0.1209(7)	0.2296(6)
S(5)	0.2304(3)	−0.0346(6)	0.2198(5)
O(1)	0.0035(13)	0.2977(33)	0.0368(21)
O(2)	0.1074(12)	0.3648(33)	−0.0288(21)
O(3)	0.0302(15)	0.2858(34)	−0.1350(26)
O(4)	0.2018(8)	−0.1746(21)	0.2293(15)
O(5)	0.2607(8)	−0.0115(20)	0.1163(15)
O(6)	0.2687(7)	0.0024(17)	0.3152(14)
H₂O(1)	0.1299(9)	0.3334(25)	0.4582(16)
H₂O(2)	0	0.3852(51)	1/4

Table 3. Temperature parameters in Å². For Rb, Te, and S, the expression used is
$$\exp[-2\pi^2(h^2a^{-2}U_{11} + \cdots + 2hka^{-1}b^{-1}U_{12} + \cdots)].$$ For O, the expression used is
$$\exp[-8\pi^2U(\sin^2\theta/\lambda^2)].$$ 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}
Rb(1)	476(13)	281(11)	477(19)	−13(13)	6(14)	37(12)
Rb(2)	529(15)	583(20)	839(25)	103(16)	−13(21)	−87(16)
S(1)	472(34)	606(52)	659(58)	−43(35)	183(50)	−55(38)
S(2)	560(40)	562(46)	706(62)	−135(37)	−230(51)	70(41)
Te	475(9)	618(13)	861(20)	−134(10)	241(13)	100(11)
S(4)	602(38)	305(37)	541(53)	72(32)	−85(38)	−35(35)
S(5)	516(33)	162(29)	415(47)	−19(25)	6(31)	−8(30)

	U		U		U
O(1)	1266(91)	O(3)	594(53)	H₂O(1)	787(63)
O(2)	1177(90)	O(4)	579(53)	H₂O(2)	1424(151)
O(3)	1603(133)	O(5)	462(44)		

THE TELLUROPENTATHIONATE ION

Distances and angles in the telluropentathionate ion, calculated from the atomic coordinates of Table 2, are listed in Table 5. The standard deviations in unit cell dimensions are small and have been neglected.

Table 4. Observed and calculated structure factors for rubidium telluropentathionate hemitrihydrate. Unobserved reflections are given the value of the observable limit, and are indicated by a minus sign on F(O).

H	K	L	F(O)	F(C)
4	0	0	54	57
6	0	0	906	-781
8	0	0	120	107
10	0	0	107	-107
12	0	0	235	253
14	0	0	29	23
16	0	0	85	86
18	0	0	57	53
20	0	0	31	-29
22	0	0	73	-75
24	0	0	44	-49
26	0	0	45	55
1	1	0	206	202
3	1	0	51	45
5	1	0	33	-36
7	1	0	430	-389
9	1	0	408	-365
11	1	0	42	45
13	1	0	316	338
15	1	0	158	164
17	1	0	42	-51
19	1	0	116	-122
21	1	0	78	-81
23	1	0	-31	-2
25	1	0	28	32
0	2	0	289	259
2	2	0	205	194
4	2	0	374	-334
6	2	0	334	-322
8	2	0	252	-234
10	2	0	149	141
12	2	0	103	109
14	2	0	167	173
16	2	0	-30	-17
18	2	0	-31	11
20	2	0	115	-123
22	2	0	60	-55
24	2	0	27	-30
1	3	0	487	425
3	3	0	208	185
5	3	0	285	-262
7	3	0	260	-253
9	3	0	85	-88
11	3	0	34	38
13	3	0	126	126
15	3	0	117	123
17	3	0	41	42
19	3	0	51	-59
21	3	0	50	-52
23	3	0	-28	-18
25	3	0	-22	-9
0	4	0	449	407
2	4	0	145	-101
4	4	0	467	412
6	4	0	461	-450
8	4	0	59	-75
10	4	0	250	-248
12	4	0	157	146
14	4	0	117	107
16	4	0	132	145
18	4	0	38	-49
20	4	0	81	-83
22	4	0	-29	-12
24	4	0	-23	-19
1	5	0	172	178
3	5	0	-29	-14
5	5	0	220	-226
7	5	0	187	-191
9	5	0	70	63
11	5	0	80	87
13	5	0	47	16
15	5	0	50	52
17	5	0	41	35
19	5	0	39	-24
21	5	0	59	-62
23	5	0	48	-53
0	6	0	65	-69
2	6	0	124	122
4	6	0	-32	-15
6	6	0	50	47
8	6	0	216	-217
10	6	0	-34	-15
12	6	0	-32	-6
14	6	0	163	185
16	6	0	-35	32
18	6	0	-34	-29
20	6	0	64	-86
22	6	0	-23	-18
1	7	0	159	177
3	7	0	-34	6
5	7	0	75	-97
7	7	0	57	-67
9	7	0	71	-70
11	7	0	-37	16
13	7	0	-38	11
15	7	0	55	48
17	7	0	39	41
19	7	0	-28	29
21	7	0	38	-53
0	8	0	177	185
2	8	0	79	-81
4	8	0	86	94
6	8	0	119	-110
8	8	0	-37	-6
10	8	0	116	-133
12	8	0	107	113
14	8	0	-37	35
16	8	0	36	38
18	8	0	-25	-17
1	9	0	-32	36
3	9	0	-33	-15
5	9	0	36	-35
7	9	0	-35	7
9	9	0	63	-61
11	9	0	-35	17
13	9	0	42	44
15	9	0	33	44
0	10	0	-31	-7
2	10	0	98	118
4	10	0	86	-103
6	10	0	-31	21
8	10	0	66	-86
10	10	0	36	42
12	10	0	-24	-10
1	11	0	-25	12
3	11	0	28	42
5	11	0	-24	-19
7	11	0	-22	-24
1	1	1	107	107
2	1	1	111	-113
3	1	1	24	-17
4	1	1	105	-97
5	1	1	69	-69
6	1	1	248	-232
7	1	1	146	-136
8	1	1	69	59
9	1	1	163	147
10	1	1	53	-55
11	1	1	-19	-7
12	1	1	142	147
13	1	1	73	-73
14	1	1	80	-83
15	1	1	45	-43
16	1	1	93	94
17	1	1	28	29
18	1	1	53	-64
19	1	1	-24	4
20	1	1	52	57
21	1	1	24	24
22	1	1	59	-59
23	1	1	-23	-20
24	1	1	-21	21
25	1	1	-18	-2
26	1	1	33	-42
0	2	1	188	-208
1	2	1	76	-74
2	2	1	82	-82
3	2	1	107	-108
4	2	1	250	285
5	2	1	65	57
6	2	1	24	26
7	2	1	93	96
8	2	1	57	-49
9	2	1	41	41
10	2	1	35	-23
11	2	1	-21	-9
12	2	1	-21	-6
13	2	1	34	-35
14	2	1	85	85
15	2	1	-22	-17
16	2	1	36	29
17	2	1	-23	0
18	2	1	73	-76
19	2	1	-24	5
20	2	1	41	-38
21	2	1	-25	-1
22	2	1	55	49
23	2	1	-22	6
24	2	1	26	24
25	2	1	-18	5
1	3	1	62	64
2	3	1	55	53
3	3	1	80	-73
4	3	1	35	-27
5	3	1	185	-199
6	3	1	104	102
7	3	1	43	-34
8	3	1	46	-54
9	3	1	205	222
10	3	1	142	159
11	3	1	67	63
12	3	1	84	-79
13	3	1	95	-94
14	3	1	24	31
15	3	1	69	-68
16	3	1	130	-139
17	3	1	-24	20
18	3	1	56	58
19	3	1	-24	23
20	3	1	-25	-26
21	3	1	-25	2
22	3	1	75	72
23	3	1	-21	-7
24	3	1	31	-31
25	3	1	-16	-12
0	4	1	123	-118
1	4	1	60	-51
2	4	1	64	-58
3	4	1	193	-181
4	4	1	181	173
5	4	1	73	-69
6	4	1	21	22
7	4	1	-20	6
8	4	1	-21	-19
9	4	1	76	71
10	4	1	93	-89
11	4	1	-24	-9
12	4	1	50	45
13	4	1	-25	-9
14	4	1	48	42
15	4	1	-25	10
16	4	1	-25	-26
17	4	1	31	26
18	4	1	41	-37
19	4	1	-25	2
20	4	1	42	-30
21	4	1	26	-19
22	4	1	51	50
23	4	1	-20	-11
24	4	1	-17	17
1	5	1	75	66
2	5	1	66	-66
3	5	1	78	-84
4	5	1	-22	-22
5	5	1	-22	-10
6	5	1	114	-104
7	5	1	-22	16
8	5	1	63	64
9	5	1	87	85
10	5	1	34	-36
11	5	1	40	-36
12	5	1	124	106
13	5	1	35	32
14	5	1	54	-56
15	5	1	101	-91
16	5	1	44	48
17	5	1	33	34
18	5	1	60	-57
19	5	1	-25	-3
20	5	1	65	54
21	5	1	-22	14
22	5	1	41	-44
23	5	1	-17	-8
0	6	1	85	90
1	6	1	-24	7
2	6	1	120	-115
3	6	1	-24	-9
4	6	1	-24	4
5	6	1	24	23
6	6	1	-24	8
7	6	1	52	39
8	6	1	60	52
9	6	1	43	24
10	6	1	-26	-9
11	6	1	-26	-6
12	6	1	51	44
13	6	1	-27	-18
14	6	1	-28	-13
15	6	1	-27	-10
16	6	1	69	-63
17	6	1	-26	-18
18	6	1	-26	7
19	6	1	-24	2
20	6	1	-22	3
21	6	1	-20	1
22	6	1	-17	15
1	7	1	33	-35
2	7	1	-25	10
3	7	1	55	-51
4	7	1	57	-53
5	7	1	106	96
6	7	1	50	49
7	7	1	-26	21
8	7	1	39	-38
9	7	1	-27	-4
10	7	1	67	63
11	7	1	34	-32
12	7	1	83	-75
13	7	1	61	48
14	7	1	64	57
15	7	1	-28	-21
16	7	1	54	-42
17	7	1	27	-22
18	7	1	49	51
19	7	1	-21	-12
20	7	1	48	-47
21	7	1	-15	2
0	8	1	92	100
1	8	1	31	27
2	8	1	53	-53
3	8	1	-25	-25
4	8	1	-26	-13
5	8	1	-26	-10
6	8	1	47	-42
7	8	1	63	-60
8	8	1	72	68
9	8	1	-28	-6
10	8	1	36	32
11	8	1	-28	15
12	8	1	-29	-11
13	8	1	58	44
14	8	1	51	-38
15	8	1	-26	16
16	8	1	39	-34
17	8	1	-21	0
18	8	1	23	24
19	8	1	-16	-9
1	9	1	51	-59
2	9	1	26	-24
3	9	1	-25	15
4	9	1	-25	21
5	9	1	60	68
6	9	1	-26	-17
7	9	1	-26	-23
8	9	1	40	33
9	9	1	29	28
10	9	1	52	-48
11	9	1	45	-45
12	9	1	39	34
13	9	1	45	40
14	9	1	-22	-25
15	9	1	-19	-16
16	9	1	50	52
1	0	2	161	-180
2	0	2	28	25
3	0	2	379	-375
4	0	2	226	239
5	0	2	85	-83
6	0	2	146	-130
7	0	2	98	96
8	0	2	109	96
9	0	2	258	255
10	0	2	144	-162
11	0	2	98	97
12	0	2	59	50
13	0	2	37	35
14	0	2	48	-48
15	0	2	55	-45
16	0	2	76	76
17	0	2	52	-52
18	0	2	71	-81
19	0	2	90	-97
20	0	2	45	39
21	0	2	31	-37
22	0	2	61	60
23	0	2	46	49
24	0	2	34	36
25	0	2	66	73
26	0	2	31	-39
2	1	2	150	-157
3	1	2	52	45
4	1	2	192	-200
5	1	2	183	-163
6	1	2	255	-247
7	1	2	53	52
8	1	2	-17	16
9	1	2	159	151
10	1	2	216	216
11	1	2	108	100
12	1	2	248	253
13	1	2	125	-129
14	1	2	-20	-2
15	1	2	58	-57
16	1	2	119	-126
17	1	2	-22	-13
18	1	2	115	-122
19	1	2	39	38
20	1	2	-23	-20
21	1	2	24	23
22	1	2	41	42
23	1	2	-22	6
24	1	2	43	43
25	1	2	-18	-14
26	1	2	19	23
0	2	2	629	-580
1	2	2	341	-330
2	2	2	188	163
3	2	2	269	-274
4	2	2	245	-237
5	2	2	351	-355
6	2	2	321	319
7	2	2	119	109
8	2	2	180	-170
9	2	2	74	69
10	2	2	165	173
11	2	2	206	208
12	2	2	79	-76
13	2	2	35	36
14	2	2	26	33
15	2	2	-22	14
16	2	2	80	-79
17	2	2	90	-95
18	2	2	49	-47
19	2	2	73	-76
20	2	2	-24	13
21	2	2	24	-27
22	2	2	-24	12
23	2	2	28	36
24	2	2	26	34
25	2	2	49	52
1	3	2	75	64
2	3	2	158	-155
3	3	2	102	-87
4	3	2	231	-229
5	3	2	35	36
6	3	2	114	-114
7	3	2	103	108
8	3	2	124	135
9	3	2	-20	-8
10	3	2	169	178
11	3	2	-22	-8
12	3	2	126	131
13	3	2	69	-66
14	3	2	-23	-24
15	3	2	-23	-6
16	3	2	78	-80
17	3	2	-24	-17
18	3	2	90	-96
19	3	2	54	54
20	3	2	28	-26
21	3	2	24	17
22	3	2	29	24
23	3	2	-21	12
24	3	2	45	45
25	3	2	-15	0
0	4	2	201	200
1	4	2	-19	-1
2	4	2	142	-137
3	4	2	200	-200
4	4	2	99	87
5	4	2	117	-119
6	4	2	-19	-7
7	4	2	47	-46
8	4	2	139	135
9	4	2	169	164
10	4	2	91	-92
11	4	2	141	136
12	4	2	53	47
13	4	2	86	87
14	4	2	108	-113
15	4	2	80	-80
16	4	2	-25	7
17	4	2	110	-113
18	4	2	26	28
19	4	2	62	-61
20	4	2	77	81
21	4	2	27	28
22	4	2	-21	-17
23	4	2	43	45
24	4	2	-16	-4
1	5	2	174	-185
2	5	2	249	-257
3	5	2	50	-36
4	5	2	243	-243
5	5	2	114	116
6	5	2	50	-48
7	5	2	114	119
8	5	2	144	135
9	5	2	72	-62
10	5	2	107	97
11	5	2	33	-32
12	5	2	53	52
13	5	2	-26	18
14	5	2	-26	2
15	5	2	30	-20
16	5	2	43	-31
17	5	2	31	-27
18	5	2	62	-57
19	5	2	-25	-2
20	5	2	39	-37
21	5	2	-22	5
22	5	2	-19	7
23	5	2	-17	8
0	6	2	146	-158
1	6	2	87	-87

Table 4. Continued.

H	K	L	F(O)	F(C)
2	6	2	133	129
3	6	2	44	-35
4	6	2	228	-202
5	6	2	203	-193
6	6	2	167	158
7	6	2	26	-20
8	6	2	26	20
9	6	2	66	58
10	6	2	107	107
11	6	2	181	181
12	6	2	112	-104
13	6	2	38	37
14	6	2	37	-36
15	6	2	52	-44
16	6	2	39	-31
17	6	2	109	-101
18	6	2	56	54
19	6	2	27	-25
20	6	2	21	25
21	6	2	-19	17
22	6	2	16	-13
1	7	2	78	-84
2	7	2	138	-147
3	7	2	72	69
4	7	2	114	-108
5	7	2	103	92
6	7	2	-25	7
7	7	2	-26	16
8	7	2	89	86
9	7	2	30	-35
10	7	2	82	69
11	7	2	63	-57
12	7	2	30	27
13	7	2	28	-23
14	7	2	-28	-19
15	7	2	27	20
16	7	2	42	-31
17	7	2	-24	13
18	7	2	44	-42
19	7	2	-20	9
20	7	2	23	-24
0	8	2	28	31
1	8	2	-24	4
2	8	2	102	-107
3	8	2	106	-114
4	8	2	103	110
5	8	2	26	-24
6	8	2	78	-76
7	8	2	37	-41
8	8	2	104	100
9	8	2	94	82
10	8	2	30	-30
11	8	2	75	63
12	8	2	28	-18
13	8	2	30	29
14	8	2	65	-62
15	8	2	56	-50
16	8	2	43	45
17	8	2	41	-39
18	8	2	-18	3
1	9	2	33	-48
2	9	2	50	-56
3	9	2	-24	1
4	9	2	60	-66
5	9	2	27	33
6	9	2	25	-27
7	9	2	-26	-3
8	9	2	-26	12
9	9	2	65	55
10	9	2	66	56
11	9	2	26	-18
12	9	2	43	39
13	9	2	-23	-16
14	9	2	-21	-3
15	9	2	-18	-12
16	9	2	31	-29
0	10	2	74	-87
1	10	2	45	-47
2	10	2	34	41
3	10	2	42	-41
4	10	2	-23	-15
5	10	2	40	-45
6	10	2	55	63
7	10	2	32	33
8	10	2	43	-42
9	10	2	21	21
10	10	2	-20	17
11	10	2	31	37
12	10	2	36	-43
13	10	2	-15	-9
1	11	2	-18	19
2	11	2	-18	-1
3	11	2	-18	-18
4	11	2	17	-22
5	11	2	-17	3
6	11	2	17	-16
7	11	2	-16	-5
8	11	2	-15	2
1	1	3	74	77
2	1	3	153	-169
3	1	3	31	26
4	1	3	62	-64
5	1	3	38	22
6	1	3	56	-53
7	1	3	50	-51
8	1	3	201	201
9	1	3	82	-84
10	1	3	90	-95
11	1	3	69	-64
12	1	3	30	30
13	1	3	90	91
14	1	3	58	-58
15	1	3	51	-53
16	1	3	109	118
17	1	3	30	33
18	1	3	28	-34
19	1	3	25	-19
20	1	3	32	37
21	1	3	26	30
22	1	3	57	-64
23	1	3	-23	-14
24	1	3	-21	22
25	1	3	-18	20
0	2	3	129	126
1	2	3	81	78
2	2	3	30	22
3	2	3	176	182
4	2	3	183	-190
5	2	3	60	-57
6	2	3	-17	7
7	2	3	77	-75
8	2	3	42	44
9	2	3	-20	-11
10	2	3	-21	2
11	2	3	71	64
12	2	3	64	56
13	2	3	31	27
14	2	3	-23	-18
15	2	3	73	-70
16	2	3	32	-31
17	2	3	72	-64
18	2	3	-25	9
19	2	3	33	30
20	2	3	-26	5
21	2	3	67	66
22	2	3	25	-24
23	2	3	-23	7
24	2	3	-20	7
25	2	3	20	-23
1	3	3	-18	-17
2	3	3	146	-144
3	3	3	-17	-3
4	3	3	116	-106
5	3	3	-18	-2
6	3	3	192	216
7	3	3	100	95
8	3	3	109	110
9	3	3	107	-114
10	3	3	-23	2
11	3	3	117	116
12	3	3	196	-207
13	3	3	25	26
14	3	3	-25	-7
15	3	3	58	64
16	3	3	-25	-18
17	3	3	86	-102
18	3	3	67	78
19	3	3	-26	11
20	3	3	35	-37
21	3	3	42	-39
22	3	3	29	32
23	3	3	30	38
24	3	3	-19	-17
0	4	3	96	95
1	4	3	48	42
2	4	3	63	63
3	4	3	100	87
4	4	3	94	-95
5	4	3	76	-71
6	4	3	46	-50
7	4	3	63	-59
8	4	3	50	-51
9	4	3	-23	8
10	4	3	52	48
11	4	3	82	73
12	4	3	-26	-5
13	4	3	-26	2
14	4	3	-27	-17
15	4	3	50	-55
16	4	3	-26	-4
17	4	3	-26	-10
18	4	3	-26	-3
19	4	3	36	31
20	4	3	-26	20
21	4	3	52	51
22	4	3	-22	-18
23	4	3	-20	-12
24	4	3	-17	8
1	5	3	-23	19
2	5	3	94	-104
3	5	3	31	35
4	5	3	49	32
5	5	3	-23	-10
6	5	3	-23	3
7	5	3	76	-64
8	5	3	108	100
9	5	3	-25	-8
10	5	3	98	-91
11	5	3	-26	-3
12	5	3	50	36
13	5	3	-27	13
14	5	3	96	-94
15	5	3	-28	20
16	5	3	82	85
17	5	3	-27	-2
18	5	3	29	-29
19	5	3	34	-34
20	5	3	54	51
21	5	3	26	24
22	5	3	23	-30
23	5	3	-17	-2
0	6	3	77	-79
1	6	3	63	-60
2	6	3	57	53
3	6	3	57	53
4	6	3	-25	-13
5	6	3	33	26
6	6	3	-26	-21
7	6	3	78	68
8	6	3	-26	10
9	6	3	-27	-8
10	6	3	66	58
11	6	3	-28	-16
12	6	3	-28	6
13	6	3	76	-70
14	6	3	-29	-2
15	6	3	48	-39
16	6	3	32	23
17	6	3	27	22
18	6	3	46	-39
19	6	3	24	26
20	6	3	-22	-9
21	6	3	-19	22
22	6	3	-16	-4
1	7	3	27	-25
2	7	3	59	56
3	7	3	6	57
4	7	3	58	45
5	7	3	89	-83
6	7	3	-27	13
7	7	3	43	29
8	7	3	60	-54
9	7	3	30	-23
10	7	3	37	26
11	7	3	83	81
12	7	3	32	-32
13	7	3	75	-72
14	7	3	-30	8
15	7	3	48	44
16	7	3	54	-47
17	7	3	44	-39
18	7	3	42	43
19	7	3	20	20
20	7	3	-18	-2
0	8	3	50	-54
1	8	3	51	-50
2	8	3	50	50
3	8	3	-27	-14
4	8	3	-27	-9
5	8	3	-28	8
6	8	3	-28	24
7	8	3	58	53
8	8	3	35	-40
9	8	3	-29	-10
10	8	3	-29	-14
11	8	3	55	-49
12	8	3	-30	-4
13	8	3	-29	-23
14	8	3	-28	10
15	8	3	-25	14
16	8	3	-23	18
17	8	3	44	44
18	8	3	-18	-13
1	9	3	-25	22
2	9	3	49	56
3	9	3	-26	-20
4	9	3	61	67
5	9	3	-27	-14
6	9	3	41	-46
7	9	3	-28	14
8	9	3	-28	20
9	9	3	-28	0
10	9	3	56	-56
11	9	3	-27	10
12	9	3	39	48
13	9	3	-23	-2
14	9	3	45	-50
15	9	3	-19	-11
0	10	3	-24	-17
1	10	3	-24	-14
2	10	3	-24	-5
3	10	3	39	38
4	10	3	-24	-16
5	10	3	-24	5
6	10	3	37	41
7	10	3	41	49
8	10	3	-22	-44
9	10	3	-22	-22
10	10	3	-20	-11
11	10	3	26	-36
12	10	3	-17	22
2	0	4	261	-299
3	0	4	273	337
4	0	4	102	106
5	0	4	188	219
6	0	4	63	59
7	0	4	263	-282
8	0	4	227	255
9	0	4	71	-71
10	0	4	27	-25
11	0	4	105	-97
12	0	4	-21	-6
13	0	4	52	50
14	0	4	118	-119
15	0	4	49	-42
16	0	4	69	-64
17	0	4	-25	8
18	0	4	34	28
19	0	4	93	36
20	0	4	74	70
21	0	4	43	42
22	0	4	60	53
23	0	4	-24	-17
24	0	4	-21	12
25	0	4	35	-39
1	1	4	140	-168
2	1	4	49	34
3	1	4	145	-147
4	1	4	52	41
5	1	4	147	171
6	1	4	248	245
7	1	4	117	110
8	1	4	52	-47
9	1	4	136	122
10	1	4	145	-152
11	1	4	43	-45
12	1	4	208	-211
13	1	4	30	-16
14	1	4	97	99
15	1	4	141	-149
16	1	4	101	103
17	1	4	38	-35
18	1	4	78	83
19	1	4	-26	20
20	1	4	41	-43
21	1	4	65	70
22	1	4	41	-42
23	1	4	25	27
24	1	4	-21	-24
25	1	4	-18	7
0	2	4	304	-362
1	2	4	55	-37
2	2	4	19	7
3	2	4	320	339
4	2	4	119	-112
5	2	4	73	-55
6	2	4	230	258
7	2	4	-19	-14
8	2	4	82	77
9	2	4	200	-212
10	2	4	145	143
11	2	4	53	29
12	2	4	108	-102
13	2	4	-24	-7
14	2	4	70	-61
15	2	4	90	88
16	2	4	115	-121
17	2	4	-26	18
18	2	4	-26	18
19	2	4	55	52
20	2	4	-27	20
21	2	4	-27	-16
22	2	4	50	55
23	2	4	31	-34
24	2	4	-20	11
25	2	4	33	-39
1	3	4	130	-130
2	3	4	152	164
3	3	4	-18	-6
4	3	4	148	160
5	3	4	84	77
6	3	4	76	66
7	3	4	114	115
8	3	4	129	-125
9	3	4	153	158
10	3	4	53	-40
11	3	4	62	-59
12	3	4	107	-99
13	3	4	80	-69
14	3	4	-26	27
15	3	4	99	-100
16	3	4	-27	16
17	3	4	-27	13
18	3	4	63	63
19	3	4	-27	15
20	3	4	-27	-10
21	3	4	67	70
22	3	4	-24	-7
23	3	4	30	33
24	3	4	-18	-15
0	4	4	109	-117
1	4	4	91	78
2	4	4	193	-198
3	4	4	89	-62
4	4	4	195	191
5	4	4	241	249
6	4	4	30	-17
7	4	4	28	-12
8	4	4	190	177
9	4	4	-25	6
10	4	4	28	-20
11	4	4	168	-163
12	4	4	56	-43
13	4	4	38	-36
14	4	4	110	-114
15	4	4	-28	-2
16	4	4	-28	-13
17	4	4	64	91
18	4	4	-28	3
19	4	4	-27	18
20	4	4	47	41
21	4	4	26	-19
22	4	4	54	60
23	4	4	-19	-15
1	5	4	157	-160
2	5	4	137	135
3	5	4	26	-27
4	5	4	88	78
5	5	4	123	104
6	5	4	-25	17
7	5	4	96	79
8	5	4	156	-145
9	5	4	150	140
10	5	4	35	14
11	5	4	39	-31
12	5	4	-28	18
13	5	4	92	-81
14	5	4	-29	30
15	5	4	85	-72
16	5	4	-28	-16
17	5	4	-28	1
18	5	4	45	37
19	5	4	-27	4
20	5	4	27	18
21	5	4	44	42
22	5	4	-19	5
0	6	4	189	-213
1	6	4	134	-119
2	6	4	-26	26
3	6	4	71	49
4	6	4	68	-46
5	6	4	66	-43
6	6	4	180	179
7	6	4	158	140
8	6	4	-28	17
9	6	4	98	-78
10	6	4	100	82
11	6	4	34	-36
12	6	4	118	-103
13	6	4	32	-30
14	6	4	59	57
15	6	4	76	73
16	6	4	59	-51
17	6	4	53	53
18	6	4	28	21
19	6	4	24	20
20	6	4	29	26
21	6	4	25	-33
1	7	4	71	-68
2	7	4	99	104
3	7	4	32	-30
4	7	4	59	57
5	7	4	91	75
6	7	4	-29	4
7	7	4	101	85
8	7	4	32	-35
9	7	4	-30	20
10	7	4	-30	-13
11	7	4	43	-37
12	7	4	-31	-16
13	7	4	65	-51
14	7	4	-31	-8
15	7	4	50	-46
16	7	4	30	-22
17	7	4	-25	7
18	7	4	-23	17
19	7	4	35	38
20	7	4	29	32
0	8	4	-26	-10
1	8	4	61	55
2	8	4	99	-109
3	8	4	36	-28
4	8	4	60	52
5	8	4	65	63
6	8	4	52	48
7	8	4	-30	-7
8	8	4	105	93
9	8	4	37	17
10	8	4	47	-47
11	8	4	90	-87
12	8	4	-31	8
13	8	4	-30	14
14	8	4	68	-59
15	8	4	-25	8
16	8	4	28	-19
17	8	4	26	33
1	9	4	49	-52
2	9	4	-27	21
3	9	4	31	-33
4	9	4	-27	0
5	9	4	36	37
6	9	4	-28	-8
7	9	4	78	80
8	9	4	-29	2
9	9	4	32	12
10	9	4	-28	-18
11	9	4	-28	-12
12	9	4	-25	-19
13	9	4	-23	-20
14	9	4	-20	11

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)
15	9	4	29	-33	21	4	5	33	-30	3	0	6	26	10	13	4	6	33	23	6	10	6	-19	-19
0	10	4	74	-94	22	4	5	-17	6	4	0	6	60	-62	14	4	6	82	76	7	10	6	36	43
1	10	4	40	-42	23	4	5	-14	11	5	0	6	277	320	15	4	6	50	-47	8	10	6	24	-28
2	10	4	40	38	1	5	5	73	76	6	0	6	268	-306	16	4	6	37	29	9	10	6	32	-34
3	10	4	83	97	2	5	5	-20	11	7	0	6	66	-72	17	4	6	30	25	1	1	7	83	93
4	10	4	42	-48	3	5	5	32	-26	8	0	6	42	-35	18	4	6	35	-28	2	1	7	23	-30
5	10	4	45	-40	4	5	5	44	-37	9	0	6	-18	10	19	4	6	-24	17	3	1	7	36	-43
6	10	4	72	90	5	5	5	25	25	10	0	6	27	27	20	4	6	40	-39	4	1	7	-16	-6
7	10	4	25	28	6	5	5	70	-56	11	0	6	211	-226	21	4	6	45	47	5	1	7	29	-35
8	10	4	-22	-14	7	5	5	80	-81	12	0	6	82	75	22	4	6	31	-28	6	1	7	27	-22
9	10	4	48	-58	8	5	5	29	28	13	0	6	41	-30	1	5	6	125	120	7	1	7	82	-81
10	10	4	22	26	9	5	5	-22	3	14	0	6	-22	12	2	5	6	34	-28	8	1	7	64	62
11	10	4	24	21	10	5	5	38	36	15	0	6	27	-21	3	5	6	31	-25	9	1	7	47	43
1	1	5	11	4	11	5	5	88	-86	16	0	6	54	48	4	5	6	29	16	10	1	7	56	-58
2	1	5	19	13	12	5	5	54	40	17	0	6	115	119	5	5	6	118	-119	11	1	7	65	-57
3	1	5	-10	-4	13	5	5	93	98	18	0	6	-24	-9	6	5	6	34	24	12	1	7	-24	-6
4	1	5	24	18	14	5	5	-24	-17	19	0	6	-25	-9	7	5	6	63	-50	13	1	7	45	45
5	1	5	34	24	15	5	5	52	-44	20	0	6	33	-32	8	5	6	77	66	14	1	7	-26	1
6	1	5	81	-82	16	5	5	31	-29	21	0	6	-24	-13	9	5	6	43	-33	15	1	7	89	-93
7	1	5	171	-201	17	5	5	63	66	22	0	6	36	-27	10	5	6	92	-88	16	1	7	27	25
8	1	5	23	-25	18	5	5	35	-27	23	0	6	-19	-6	11	5	6	58	53	17	1	7	65	64
9	1	5	28	19	19	5	5	36	-34	24	0	6	-16	-17	12	5	6	60	-48	18	1	7	-28	-18
10	1	5	23	15	20	5	5	38	35	1	1	6	72	77	13	5	6	58	46	19	1	7	-28	-6
11	1	5	95	-94	21	5	5	30	32	2	1	6	122	137	14	5	6	-27	-2	20	1	7	-26	11
12	1	5	108	105	22	5	5	-14	-5	3	1	6	110	121	15	5	6	-27	18	21	1	7	45	49
13	1	5	51	47	0	6	5	83	83	4	1	6	213	241	16	5	6	70	62	22	1	7	-22	-14
14	1	5	60	-55	1	6	5	37	38	5	1	6	75	-77	17	5	6	-26	-3	0	2	7	58	-68
15	1	5	29	-30	2	6	5	88	-40	6	1	6	70	-59	18	5	6	37	26	1	2	7	33	31
16	1	5	28	18	3	6	5	52	-53	7	1	6	124	-135	19	5	6	-22	-5	2	2	7	27	-28
17	1	5	73	68	4	6	5	-22	0	8	1	6	80	-76	20	5	6	-20	18	3	2	7	67	77
18	1	5	38	-36	5	6	5	-22	-10	9	1	6	128	-129	21	5	6	35	-34	4	2	7	-19	24
19	1	5	43	-43	6	6	5	-22	-1	10	1	6	123	-121	0	6	6	-24	-6	5	2	7	69	-68
20	1	5	50	37	7	6	5	26	16	11	1	6	54	51	1	6	6	46	-42	6	2	7	68	75
21	1	5	54	50	8	6	5	-23	13	12	1	6	67	58	2	6	6	91	82	7	2	7	-22	-12
22	1	5	-20	-12	9	6	5	47	39	13	1	6	96	92	3	6	6	151	160	8	2	7	-24	-14
23	1	5	31	-25	10	6	5	-24	-19	14	1	6	44	-41	4	6	6	36	-25	9	2	7	-25	15
24	1	5	-16	11	11	6	5	-24	0	15	1	6	115	116	5	6	6	83	-71	10	2	7	54	-52
0	2	5	92	102	12	6	5	37	32	16	1	6	54	49	6	6	6	-26	-1	11	2	7	-26	6
1	2	5	99	-96	13	6	5	-25	-1	17	1	6	-24	-17	7	6	6	-26	22	12	2	7	53	44
2	2	5	40	37	14	6	5	-25	16	18	1	6	27	14	8	6	6	104	-101	13	2	7	34	27
3	2	5	149	-169	15	6	5	-24	-3	19	1	6	70	-67	9	6	6	74	-63	14	2	7	50	43
4	2	5	22	17	16	6	5	-23	-10	20	1	6	47	42	10	6	6	30	-27	15	2	7	-27	-6
5	2	5	61	66	17	6	5	45	-43	21	1	6	52	-45	11	6	6	43	43	16	2	7	-28	-20
6	2	5	88	-82	18	6	5	-20	11	22	1	6	-22	-12	12	6	6	38	29	17	2	7	-28	-23
7	2	5	96	96	19	6	5	-18	3	23	1	6	-19	5	13	6	6	55	-43	18	2	7	47	-40
8	2	5	25	29	20	6	5	-16	-10	24	1	6	20	-19	14	6	6	96	84	19	2	7	-28	3
9	2	5	27	25	1	7	5	33	28	0	2	6	111	128	15	6	6	-27	4	20	2	7	-26	-15
10	2	5	81	81	2	7	5	30	-24	1	2	6	118	-136	16	6	6	-26	16	21	2	7	-23	16
11	2	5	42	-26	3	7	5	31	-19	2	2	6	145	165	17	6	6	-24	-15	22	2	7	-21	14
12	2	5	-20	-4	4	7	5	91	-97	3	2	6	158	189	18	6	6	-22	-20	23	2	7	-18	-14
13	2	5	66	-61	5	7	5	-23	-8	4	2	6	181	-152	19	6	6	43	44	1	3	7	63	64
14	2	5	-20	3	6	7	5	61	56	5	2	6	53	-47	20	6	6	33	-38	2	3	7	30	-32
15	2	5	-21	11	7	7	5	-24	10	6	2	6	104	-107	1	7	6	76	67	3	3	7	51	-50
16	2	5	-21	12	8	7	5	-24	-1	7	2	6	124	133	2	7	6	28	-27	4	3	7	43	-42
17	2	5	40	34	9	7	5	33	-37	8	2	6	141	-146	3	7	6	31	19	5	3	7	111	-116
18	2	5	25	-14	10	7	5	68	67	9	2	6	165	-172	4	7	6	63	56	6	3	7	-23	17
19	2	5	-22	-9	11	7	5	-25	20	10	2	6	75	64	5	7	6	71	-66	7	3	7	88	89
20	2	5	-22	-8	12	7	5	75	-71	11	2	6	-23	-19	6	7	6	65	67	8	3	7	60	51
21	2	5	38	-31	13	7	5	-25	-9	12	2	6	51	40	7	7	6	58	-50	9	3	7	34	44
22	2	5	-19	8	14	7	5	48	46	13	2	6	58	-47	8	7	6	32	30	10	3	7	31	-27
23	2	5	-17	13	15	7	5	-23	20	14	2	6	88	81	9	7	6	-28	-8	11	3	7	123	129
24	2	5	-15	-9	16	7	5	38	-39	15	2	6	100	96	10	7	6	63	-50	12	3	7	-29	-21
1	3	5	32	-27	17	7	5	-19	-17	16	2	6	30	20	11	7	6	-29	22	13	3	7	71	-73
2	3	5	85	83	18	7	5	24	29	17	2	6	62	54	12	7	6	61	-53	14	3	7	-28	28
3	3	5	61	67	0	8	5	44	40	18	2	6	-25	-13	13	7	6	-29	10	15	3	7	-28	-8
4	3	5	-16	-5	1	8	5	50	50	19	2	6	29	18	14	7	6	46	-44	16	3	7	-28	24
5	3	5	29	-35	2	8	5	-22	-4	20	2	6	46	-39	15	7	6	35	29	17	3	7	64	-71
6	3	5	24	-26	3	8	5	26	-25	21	2	6	-23	-14	16	7	6	33	28	18	3	7	-28	-6
7	3	5	-18	3	4	8	5	27	25	22	2	6	44	-39	17	7	6	-20	7	19	3	7	62	60
8	3	5	115	-124	5	8	5	-23	-5	23	2	6	33	-32	18	7	6	21	23	20	3	7	-25	-19
9	3	5	-19	-22	6	8	5	-24	-3	1	3	6	150	161	0	8	6	57	45	21	3	7	30	-30
10	3	5	139	144	7	8	5	63	-58	2	3	6	119	141	1	8	6	90	88	22	3	7	-19	-9
11	3	5	68	71	8	8	5	30	-28	3	3	6	-18	7	2	8	6	-25	-14	0	4	7	63	-53
12	3	5	-22	14	9	8	5	29	21	4	3	6	117	119	3	8	6	70	-51	1	4	7	-25	-8
13	3	5	-22	-19	10	8	5	53	-48	5	3	6	102	-102	4	8	6	53	46	2	4	7	-25	5
14	3	5	-22	19	11	8	5	45	40	6	3	6	-20	0	5	8	6	114	115	3	4	7	36	34
15	3	5	62	69	12	8	5	31	-23	7	3	6	105	-104	6	8	6	83	-75	4	4	7	33	26
16	3	5	131	-126	13	8	5	35	24	8	3	6	41	31	7	8	6	70	-72	5	4	7	55	-49
17	3	5	54	-55	14	8	5	-21	25	9	3	6	106	-93	8	8	6	-28	-10	6	4	7	-25	27
18	3	5	38	28	15	8	5	-19	-12	10	3	6	126	-118	9	8	6	40	34	7	4	7	-26	-13
19	3	5	24	-9	16	8	5	-17	18	11	3	6	63	57	10	8	6	-28	-12	8	4	7	71	-63
20	3	5	-22	-5	1	9	5	-22	15	12	3	6	44	-29	11	8	6	49	-44	9	4	7	-27	-19
21	3	5	38	-41	2	9	5	54	-54	13	3	6	70	63	12	8	6	32	28	10	4	7	33	-24
22	3	5	59	58	3	9	5	-22	-5	14	3	6	67	-59	13	8	6	-24	1	11	4	7	38	36
23	3	5	32	36	4	9	5	-22	-16	15	3	6	77	64	14	8	6	25	25	12	4	7	-30	5
0	4	5	73	84	5	9	5	26	19	16	3	6	61	54	15	8	6	32	-38	13	4	7	-30	13
1	4	5	71	-67	6	9	5	27	26	17	3	6	-25	-17	16	8	6	23	24	14	4	7	-30	20
2	4	5	43	47	7	9	5	39	-45	18	3	6	-26	8	1	9	6	-24	12	15	4	7	-29	-10
3	4	5	162	-176	8	9	5	33	22	19	3	6	-25	-16	2	9	6	-25	3	16	4	7	-29	-19
4	4	5	-18	-25	9	9	5	39	42	20	3	6	44	-43	3	9	6	-25	18	17	4	7	-28	2
5	4	5	-18	7	10	9	5	-21	4	21	3	6	47	-40	4	9	6	-25	16	18	4	7	-27	-23
6	4	5	102	-108	11	9	5	47	-54	22	3	6	-19	-12	5	9	6	-25	-13	19	4	7	-25	-9
7	4	5	72	59	12	9	5	-19	-11	23	3	6	-17	-14	6	9	6	-25	14	20	4	7	-23	11
8	4	5	35	-41	13	9	5	22	29	0	4	6	187	181	7	9	6	42	-38	21	4	7	-20	12
9	4	5	79	67	0	10	5	-18	32	1	4	6	204	208	8	9	6	34	-27	1	5	7	41	29
10	4	5	-21	-7	1	10	5	-19	12	2	4	6	-21	5	9	9	6	-23	-1	2	5	7	51	-45
11	4	5	49	-41	2	10	5	-19	-15	3	4	6	-21	-7	10	9	6	-22	-6	3	5	7	33	-28
12	4	5	29	13	3	10	5	-19	-3	4	4	6	49	32	11	9	6	-21	4	4	5	7	-27	22
13	4	5	33	-19	4	10	5	35	40	5	4	6	150	158	12	9	6	-19	-13	5	5	7	-27	-1
14	4	5	-23	-4	5	10	5	-18	11	6	4	6	89	-80	13	9	6	-16	14	6	5	7	-27	-18
15	4	5	28	23	6	10	5	19	-19	7	4	6	87	-70	0	10	6	-20	14	7	5	7	47	-38
16	4	5	-22	-1	7	10	5	27	-33	8	4	6	79	-69	1	10	6	28	-27	8	5	7	-28	27
17	4	5	26	12	8	10	5	-17	9	9	4	6	39	34	2	10	6	28	33	9	5	7	79	79
18	4	5	32	30	9	10	5	-16	7	10	4	6	92	-79	3	10	6	32	39	10	5	7	40	-36
19	4	5	-22	-2	10	10	5	-15	-10	11	4	6	135	-134	4	10	6	-20	-19	11	5	7	46	-41
20	4	5	-21	2	2	0	6	112	124	12	4	6	97	92	5	10	6	-19	-2					

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)
12	5	7	52	44	11	7	7	39	35	4	0	8	86	77	19	0	10	27	-27	6	1	8	-28	-13
13	5	7	-31	9	12	7	7	54	41	5	0	8	64	62	20	0	10	25	16	7	1	8	-29	-28
14	5	7	40	-41	13	7	7	53	-59	6	0	8	48	46	0	0	12	76	-85	8	1	8	-30	12
15	5	7	64	-59	14	7	7	-27	-5	7	0	8	121	-112	1	0	12	95	100	9	1	8	-31	29
16	5	7	48	44	15	7	7	35	30	8	0	8	72	-70	2	0	12	45	46	10	1	8	45	77
17	5	7	37	37	16	7	7	-22	6	9	0	8	173	164	3	0	12	30	-37	11	1	8	-34	5
18	5	7	27	-23	17	7	7	36	-40	10	0	8	88	-74	4	0	12	41	-49	12	1	8	34	30
19	5	7	-23	-22	0	8	7	-28	-12	11	0	8	-26	-8	5	0	12	89	84	13	1	8	-35	-8
20	5	7	-20	0	1	8	7	52	-46	12	0	8	41	35	6	0	12	53	51	14	1	8	76	60
0	6	7	60	-59	2	8	7	-28	20	13	0	8	86	68	7	0	12	-24	-9	15	1	8	-35	-13
1	6	7	75	-71	3	8	7	-29	6	14	0	8	76	64	8	0	12	51	-41	16	1	8	40	-47
2	6	7	-29	-7	4	8	7	36	-28	15	0	8	64	-58	9	0	12	-25	11	17	1	8	-34	28
3	6	7	-29	19	5	8	7	-30	-26	16	0	8	-27	11	10	0	12	32	38	18	1	8	46	-51
4	6	7	-29	-2	6	8	7	-30	12	17	0	8	71	-62	11	0	12	97	-86	19	1	8	-30	28
5	6	7	-29	-17	7	8	7	-30	23	18	0	8	25	-25	12	0	12	-24	-6	20	1	8	27	-31
6	6	7	-30	7	8	8	7	-30	18	19	0	8	48	-36	13	0	12	-22	-6	21	1	8	-24	-13
7	6	7	-30	7	9	8	7	-30	-7	20	0	8	27	-16	14	0	12	25	25	22	1	8	-20	16
8	6	7	46	45	10	8	7	-29	23	21	0	8	-18	6	15	0	12	20	22	23	1	8	18	-28
9	6	7	-31	-14	11	8	7	-28	-2	22	0	8	-16	-5	16	0	12	-15	-16	1	1	9	-27	11
10	6	7	57	54	12	8	7	40	-34	23	0	8	27	33	17	0	12	17	21	2	1	9	-28	-7
11	6	7	39	32	13	8	7	-23	8	0	0	10	93	-94	0	0	14	28	-34	3	1	9	-28	13
12	6	7	-32	2	14	8	7	37	-37	1	0	10	42	37	1	0	14	-22	-3	4	1	9	-28	24
13	6	7	-32	1	15	8	7	-18	5	2	0	10	61	-57	2	0	14	23	19	5	1	9	48	43
14	6	7	-31	-29	1	9	7	44	-42	3	0	10	54	53	3	0	14	-21	-10	6	1	9	56	51
15	6	7	-29	-8	2	9	7	-26	12	4	0	10	26	-18	4	0	14	-21	-17	7	1	9	70	-86
16	6	7	-26	-18	3	9	7	-26	-11	5	0	10	78	71	5	0	14	8	29	8	1	9	-31	-28
17	6	7	-24	20	4	9	7	-26	9	6	0	10	57	53	6	0	14	26	29	9	1	9	-32	4
18	6	7	-22	-20	5	9	7	59	60	7	0	10	85	-89	7	0	14	30	-30	10	1	9	-32	3
19	6	7	-19	-16	6	9	7	37	-37	8	0	10	99	88	8	0	14	32	-33	11	1	9	55	-61
1	7	7	74	-71	7	9	7	-25	12	9	0	10	-25	-1	9	0	14	-18	13	12	1	9	34	-19
2	7	7	-29	-28	0	0	2	50	-45	10	0	10	78	64	10	0	14	-17	10	13	1	9	48	54
3	7	7	45	38	0	0	4	210	241	11	0	10	-26	-4	27	1	0	-15	20	14	1	9	-34	-17
4	7	7	-30	15	1	0	4	488	516	12	0	10	65	-55	1	1	2	-13	-12	15	1	9	-34	-29
5	7	7	-31	4	0	0	6	264	299	13	0	10	56	46	24	1	7	-15	7	16	1	9	-33	0
6	7	7	42	-35	1	0	6	133	143	14	0	10	83	-68	1	1	8	95	105	17	1	9	-31	35
7	7	7	54	55	0	0	8	28	-26	15	0	10	32	-35	2	1	8	35	-57	18	1	9	-29	18
8	7	7	-31	-7	1	0	8	188	232	16	0	10	48	-40	3	1	8	42	-51	19	1	9	40	-42
9	7	7	43	-27	2	0	8	33	-16	17	0	10	21	16	4	1	8	129	-141	20	1	9	-23	-6
10	7	7	-32	0	3	0	8	179	-199	18	0	10	31	30	5	1	8	75	-73	21	1	9	-19	36
																				22	1	9	-15	6

Table 5. Dimensions of the telluropentathionate ion. Standard deviations are given in parentheses.

Bond lengths and angles

S(1)−S(2) = 2.116(11) Å
S(2)−Te = 2.364(9)
∠S(1)−S(2)−Te = 103.3(4)°

S(4)−S(5) = 2.126(9) Å
Te−S(4) = 2.370(7)
∠Te−S(4)−S(5) = 102.8(3)°

∠S(2)−Te−S(4) = 100.1(3)°

S(1)−O(1) = 1.42(3) Å
S(1)−O(2) = 1.40(3)
S(1)−O(3) = 1.31(3)
∠S(2)−S(1)−O(1) = 109.6(13)°
∠S(2)−S(1)−O(2) = 105.4(12)°
∠S(2)−S(1)−O(3) = 101.3(15)°
∠O(1)−S(1)−O(2) = 119.9(17)°
∠O(1)−S(1)−O(3) = 108.1(18)°
∠O(2)−S(1)−O(3) = 110.9(18)°

S(5)−O(4) = 1.46(2) Å
S(5)−O(5) = 1.46(2)
S(5)−O(6) = 1.48(2)
∠S(4)−S(5)−O(4) = 108.7(8)°
∠S(4)−S(5)−O(5) = 105.5(8)°
∠S(4)−S(5)−O(6) = 100.9(7)°
∠O(4)−S(5)−O(5) = 113.0(11)°
∠O(4)−S(5)−O(6) = 112.2(10)°
∠O(5)−S(5)−O(6) = 115.3(10)°

Dihedral angles

S(1)S(2)Te/S(2)TeS(4) = 77.7°

S(2)TeS(4)/TeS(4)S(5) = 89.2°

TeS(2)S(1)/S(2)S(1)O(1) = 32.9°
TeS(2)S(1)/S(2)S(1)O(2) = 97.4°
TeS(2)S(1)/S(2)S(1)O(3) = 147.0°
S(2)S(1)O(1)/S(2)S(1)O(2) = 130.3°
S(2)S(1)O(1)/S(2)S(1)O(3) = 114.1°
S(2)S(1)O(2)/S(2)S(1)O(3) = 115.6°

TeS(4)S(5)/S(4)S(5)O(4) = 32.1°
TeS(4)S(5)/S(4)S(5)O(5) = 89.4°
TeS(4)S(5)/S(4)S(5)O(6) = 150.3°
S(4)S(5)O(4)/S(4)S(5)O(5) = 121.5°
S(4)S(5)O(4)/S(4)S(5)O(6) = 118.2°
S(4)S(5)O(5)/S(4)S(5)O(6) = 120.3°

Non-bonded distances

S(1)−Te = 3.517(9) Å
S(1)−S(4) = 4.278(10)
S(2)−S(4) = 3.630(10)

Te−S(5) = 3.516(6) Å
S(2)−S(5) = 4.491(10)
S(1)−S(5) = 5.769(10)

The $S-S-Te-S-S$ chain has the *trans* form. Its shape corresponds approximately to the symmetry of a twofold axis passing through the tellurium atom. Fig. 1 gives a view of the telluropentathionate ion, as seen along the approximate twofold axis, with principal bond lengths and angles, and
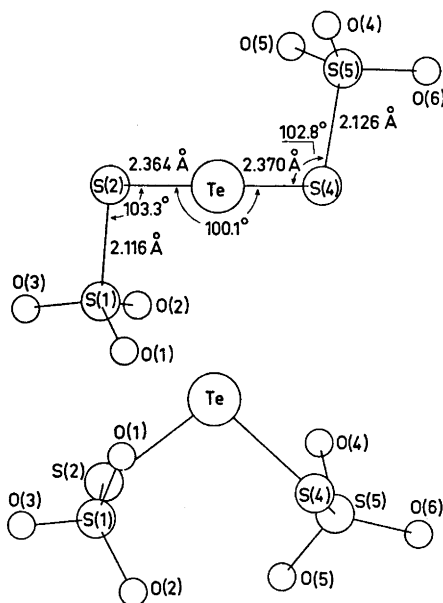

Fig. 1. The *trans* form of the telluropentathionate ion in $Rb_2Te(S_2O_3)_2.1\frac{1}{2}H_2O$, as seen along an approximate twofold axis (above), and normal to this axis and to a line through S(2) and the coordinate midpoint of the four sulphur atoms (below).

also a view normal to this axis and to a line through S(2) and the coordinate midpoint of the four sulphur atoms. The bond lengths and angles are within the errors the same in the two halves of the chain, whereas the non-bonded S(1)−S(4) and S(2)−S(5) distances are 4.278(10) Å and 4.491(9) Å, respectively. This is due to differences in the degrees of rotation about the Te−S bonds; the resulting dihedral angles, S(1)S(2)Te/S(2)TeS(4) and S(2)TeS(4)/TeS(4)S(5), differ by 11.5°.

The tellurium-sulphur bonds, between a divalent tellurium atom and a divalent sulphur atom, are 2.364(9) Å and 2.370(7) Å. The differences between these values and those found in barium telluropentathionate trihydrate,[9] 2.403(13) Å and 2.382(12) Å, in barium telluropentathionate dihydrate,[7] 2.34 Å, and in ammonium telluropentathionate,[8] 2.36 and 2.35 Å, are not significant. The standard deviations of the bond lengths in the last two compounds are estimated as 0.02 Å and 0.03 Å, respectively.[7,8] The terminal bonds, between a divalent sulphur atom and a sulphonate sulphur atom, are 2.116(11) Å and 2.126(9) Å, in accordance with the average, 2.11 Å, for this type of bond in the tetrathionate,[18] pentathionate,[2-4,11] selenopentathionate,[6,11,19] telluropentathionate,[7-9] and hexathionate[20,21] ions.

The dihedral angles, STeS/TeSS, are 78° and 89°, compared to 86° and 95° in ammonium telluropentathionate,[8] and 89° and 99° in barium telluropenta-

thionate trihydrate,[9] where the $S-S-Te-S-S$ chain has the *trans* form as in the present salt. In barium telluropentathionate dihydrate,[7] where the chain has the *cis* form, the dihedral angles are 103°, and thus larger by 14° than the average of the above values for the *trans* form.

The same tendency, of larger dihedral angles in the *cis* form than in the *trans* form, is found in selenopentathionates and pentathionates. In two barium selenopentathionates,[6,19] and in three barium pentathionates,[2-4] all having the *cis* form, the dihedral angles are 106° and 107 — 110°, respectively, compared to 83° and 85° in the *trans* form in ammonium selenopentathionate hemitrihydrate,[11] and 82° and 84° in the *trans* form in potassium pentathionate hemitrihydrate.[11] In potassium barium hexathionate, $K_2Ba(S_6O_6)_2$,[20] where the hexathionate ion has the *cis-cis* form, the dihedral angles are 23°, 16°, and 20° larger than in the *trans-trans* form of the ion, found in $[Co(en)_2Cl_2]_2S_6O_6 \cdot H_2O$.[21] The larger dihedral angles in the *cis* and *cis-cis* forms are probably due to the space requirements of the sulphonate groups.

The shape of the two thiosulphate groups, formed by the two sulphur atoms and three oxygen atoms of each half of the telluropentathionate ion, is a distorted tetrahedral one. The average $S-O$ bond length is 1.42 Å, with individual values in the range 1.31 — 1.48 Å. As seen from Table 3, the oxygen atoms O(1), O(2), and O(3), all of one thiosulphate group, have high temperature parameters, and the resulting $S-O$ distances are probably less accurate than indicated by the standard deviations listed in Table 5. The high thermal motions of the oxygen atoms may be due to an oscillation about the $S(1)-S(2)$ bond; this would cause a shortening of the observed $S-O$ bond lengths.

In each of the thiosulphate groups, there is one oxygen atom with a short distance to the tellurium atom, $Te \cdots O(1) = 3.43(3)$ Å, and $Te \cdots O(4) = 3.40(2)$ Å. The corresponding TeSS/SSO dihedral angles are 32.9° and 32.1°, respectively.

THE IONIC ENVIRONMENT

One finds, in the literature, examples of varied coordination of oxygen to rubidium. Sevenfold coordination is found in rubidium hydrogen fumarate,[22] with $Rb-O$ distances ranging from 2.88 to 3.09 Å, and in rubidium metaphosphate,[23] with $Rb-O$ distances from 2.90 to 3.20 Å. Eightfold coordination, with $Rb-O$ distances ranging from 2.89 to 3.32 Å, is found in the monorubidium salt of furantetracarboxylic acid,[24] ninefold coordination, with $Rb-O$ distances within 3.2 Å, in the rubidium salt of 5-fluoroacetic acid,[25] and fourteenfold coordination, with $Rb-O$ distances from 2.94 to 3.29 Å, in rubidium uranyl nitrate.[26]

In the present salt, the rubidium ions are each surrounded by seven oxygen atoms at distances within 3.25 Å. These approaches include all the oxygen atoms of the water molecules, and the oxygen atoms of the thiosulphate groups except O(1). The $Rb-O$ distances and related angles are listed in Table 6, and a view of the $Rb-O$ arrangement is shown in Fig. 2. The mean value of the $Rb-O$ distances listed is 3.06 Å, as compared to the sum of the ionic radii for rubidium and oxygen, 2.88 Å.[27] Each rubidium ion is in

Table 6. Distances (Å), and angles (°) between directions, from Rb(1) to oxygen atoms (above) and from Rb(2) to oxygen atoms (below). Standard deviations of the bond lengths and angles are 0.02 − 0.03 Å and 0.4 − 0.6°, respectively.

				I	II	III	IV	V	VI
I	$H_2O(1)$	(x,y,z)	2.90						
II	O(6)	(x,y,z)	3.05	103					
III	O(2)	$(\frac{1}{2}-x,\frac{1}{2}-y,\frac{1}{2}+z)$	3.01	173	71				
IV	O(5)	$(\frac{1}{2}-x,\frac{1}{2}-y,\frac{1}{2}+z)$	3.09	71	174	115			
V	O(4)	$(\frac{1}{2}-x,\frac{1}{2}+y,z)$	3.17	98	64	82	115		
VI	O(6)	$(\frac{1}{2}-x,\frac{1}{2}+y,z)$	3.11	65	102	118	74	46	
VII	O(5)	$(x,\bar{y},\frac{1}{2}+z)$	2.91	104	78	73	104	140	168

				I	II	III	IV	V	VI
I	$H_2O(1)$	$(\frac{1}{2}-x,\frac{1}{2}-y,-\frac{1}{2}+z)$	3.02						
II	$H_2O(2)$	$(\frac{1}{2}-x,-\frac{1}{2}+y,z)$	3.07	120					
III	O(5)	(x,y,z)	3.09	70	125				
IV	O(6)	(x,y,z)	3.16	114	111	47			
V	O(3)	$(\frac{1}{2}-x,\frac{1}{2}-y,\frac{1}{2}+z)$	2.99	133	70	147	102		
VI	O(3)	$(\frac{1}{2}+x,\frac{1}{2}-y,\bar{z})$	3.25	81	67	150	162	60	
VII	O(4)	$(\frac{1}{2}-x,\frac{1}{2}+y,z)$	3.06	82	155	71	64	87	110
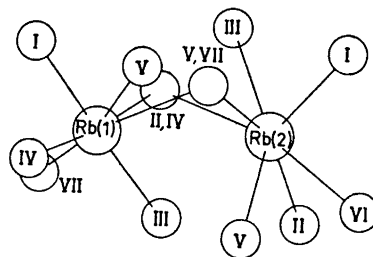
Fig. 2. A view of the rubidium-oxygen coordination in $Rb_2Te(S_2O_3)_2.1\frac{1}{2}H_2O$, as seen along the b axis. The oxygen atoms are marked I − VII in accordance with Table 6.

contact with thiosulphate oxygen atoms of four different anions. The telluropentathionate ions are thus held together by the rubidium ions. From the unsymmetrical rubidium-oxygen configuration, one might conclude that the packing in the crystal is, at least partly, controlled by the packing of the anions.

Close contacts, probably hydrogen-bonded, occur between the water molecules and the oxygen atoms of the thiosulphate groups. The approaches are: $H_2O(1) - O(1') = 2.86(3)$ Å, $H_2O(1) - O(2') = 2.90(4)$ Å, $\angle O(1') - H_2O(1) - O(2') = 87.1(10)°$, and $H_2O(2) - O(1) = 2.78(3)$ Å, $\angle O(1) - H_2O(2) - O(1') = 145.4(16)°$. Here $O(2')$ is at $x,1-y,\frac{1}{2}-z$ relative to O(2), and O(1) and O(1') are related to each other through the twofold axis passing through $H_2O(2)$. The oxygen atom O(1), not included in the rubidium oxygen approaches, is in close contact with both of the water molecules, at $H_2O(1) - O(1) - H_2O(2) = 85.3(9)°$.

The closest interionic Te−Te and Te−S approaches are, Te−Te′ = 3.925(3) Å and Te−S(2′) = 3.738(7) Å, where Te′ is at $\bar{x},y,\frac{1}{2}-z$ relative to Te, and S(2′) is at $\bar{x},\bar{y},\bar{z}$ relative to S(2). The angles between the directions from Te to S(2), S(4), Te′, and S(2′) are, $\angle$S(2)−Te−S(4) = 100°, $\angle$S(4)−Te−Te′ = 110°, $\angle$Te′−Te−S(2′) = 63°, $\angle$S(2′)−Te−S(2) = 78°, $\angle$S(2)−Te−Te′ = 135°, and $\angle$S(4)−Te−S(2′) = 163°. Te′ and S(2′) are located on the same side of the plane through Te, S(2), and S(4), at distances of 2.05 Å and 1.10 Å, respectively, from the plane.

REFERENCES

1. Foss, O. *Advan. Inorg. Chem. Radiochem.* **2** (1960) 237.
2. Foss, O. and Zachariasen, H. *Acta Chem. Scand.* **8** (1954) 473.
3. Foss, O. and Tjomsland, O. *Acta Chem. Scand.* **10** (1956) 288.
4. Foss, O. and Tjomsland, O. *Acta Chem. Scand.* **12** (1958) 44.
5. Foss, O. and Tjomsland, O. *Acta Chem. Scand.* **8** (1954) 1701.
6. Marøy, K. *Acta Chem. Scand.* **24** (1970) 3401.
7. Foss, O. and Tjomsland, O. *Acta Chem. Scand.* **12** (1958) 52.
8. Foss, O. and Larssen, P. A. *Acta Chem. Scand.* **8** (1954) 1042.
9. Gjerrestad, K. and Marøy, K. *Acta Chem. Scand.* **24** (1970) 3402.
10. Foss, O. and Jahr, J. *Acta Chem. Scand.* **4** (1950) 1560.
11. Marøy, K. *Acta Chem. Scand.* **23** (1969) 338.
12. Foss, O. *Inorg. Syn.* **4** (1953) 88.
13. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham 1962, Vol. III.
14. Coppens, P., Leiserowitz, L. and Rabinovich, D. *Acta Cryst.* **18** (1965) 1035.
15. Bøyum, K. and Marøy, K. *Acta Chem. Scand.* **25** (1971) 2569.
16. Zachariasen, W. H. *Acta Cryst.* **16** (1963) 1139.
17. Cromer, D. T. *Acta Cryst.* **18** (1965) 17.
18. Foss, O. and Hordvik, A. *Acta Chem. Scand.* **18** (1964) 662.
19. Marøy, K. *To be published.*
20. Foss, O. and Johnsen, K. *Acta Chem. Scand.* **19** (1965) 2207.
21. Foss, O. and Marøy, K. *Acta Chem. Scand.* **19** (1965) 2219.
22. Gupta, M. P. *Acta Cryst.* **9** (1956) 263.
23. Corbridge, D. E. C. *Acta Cryst.* **9** (1956) 308.
24. Paul, I. C. and Martin, L. *Acta Cryst.* **22** (1967) 559.
25. Macintyre, W. M. and Zirakzadeh, M. *Acta Cryst.* **17** (1964) 1305.
26. Barclay, G. A., Sabine, T. M. and Taylor, J. C. *Acta Cryst.* **19** (1965) 205.
27. Pauling, L. *The Nature of the Chemical Bond*, 3rd Ed., Cornell University Press, Ithaca, New York 1960.