The Crystal Structure of Ammonium Selenopentathionate

Hemitrihydrate

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The salt, $(NH_4)_2Se(S_2O_3)_2.1\frac{1}{2}H_2O$, crystallizes in the orthorhombic space group *Pbcn* (No. 60) with eight formula units per unit cell, and $a = 20.718(10)$ Å, $b = 9.326(4)$ Å, $c = 12.590(6)$ Å. The structure has been determined by X-ray methods, and refined by least squares for the $hk0 - hk7$, and $h0l$ reflections.

The selenopentathionate ion has the *trans* form in this salt, the terminal sulphur atoms being located on opposite sides of the plane through the three middle atoms. The dimensions of the $S - S - Se - S - S$ chain are: $S(1) - S(2) = 2.120(4)$ Å, $S(2) - Se = 2.153(4)$ Å, $Se - S(4) = 2.181(3)$ Å, $S(4) - S(5) = 2.116(4)$ Å, $\angle S(1) - S(2) - Se = 104.0(2)°$, $\angle S(2) - Se - S(4) = 104.9(2)°$, $\angle Se - S(4) - S(5) = 101.1(2)°$. The dihedral angles, $S(1)S(2)Se/S(2)SeS(4) = 83.3°$ and $S(2)SeS(4)/SeS(4)S(5) = 85.2°$.

Ammonium selenopentathionate hemitrihydrate belongs to a series of isomorphous alkali telluropentathionate, selenopentathionate, and pentathionate hemitrihydrates.[1] The $S - S - Te - S - S$, $S - S - Se - S - S$, and S—S—S—S—S chains of the ions have the *trans* rotational-isomeric form in these salts; the terminal sulphur atoms are located on opposite sides of the plane through the three middle atoms.[2] The *cis* form of the selenopentathionate ion, with the terminal atoms on the same side of the plane, occurs in the crystals of barium selenopentathionate dihydrate [3,4] and barium selenopentathionate trihydrate.[5] This structure analysis of ammonium selenopentathionate hemitrihydrate is the first of a salt in which the selenopentathionate ion has the *trans* form.

EXPERIMENTAL

Ammonium selenopentathionate hemitrihydrate, $(NH_4)_2Se(S_2O_3)_2.1\frac{1}{2}H_2O$, was prepared from selenious acid and ammonium thiosulphate, and recrystallized from dilute hydrochloric acid.[1]

The crystals are light yellow, 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. CuKα radiation was used, and the Weissenberg camera

radius was checked with sodium chloride, $a = 5.6394$ Å (Ref. 6, p. 122). The unit cell is orthorhombic, with axial lengths $a = 20.718(10)$ Å, $b = 9.326(4)$ Å $c = 12.590(6)$ 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), with eight formula units per unit cell.[1]

The intensities of the $hk0 - hk7$ and $h0l$ reflections were estimated visually from zero-layer and equi-inclination Weissenberg photographs, taken with $CuK\alpha$ radiation (Ni-filtered), using the multiple-film technique. Altogether 1414 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_o, 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 absorption coefficient for $CuK\alpha$ radiation is 112 cm^{-1}. No correction for absorption was made.

The atomic scattering curves used for the calculations of structure factors were those listed in *International Tables for X-ray Crystallography*,[6] Table 3.3.1A. The scattering curve for selenium was corrected for anomalous dispersion, using the $\Delta f'$ and $\Delta f''$ values calculated by Cromer,[9] and taking the amplitude of f as the corrected value.

The computer programs used for data processing and least squares refinement 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-Moe, and the program used for Fourier summations by Mr. E. Alver, both of this Institute. The calculations were carried out on an IBM 360/50H computer.

THE STRUCTURE ANALYSIS

A three-dimensional Patterson synthesis, based on the 1414 observed $hk0 - hk7$ and $h0l$ reflections, brought to the same scale by comparison with $h1l$ reflections, was calculated. The derived selenium x and y coordinates, from the Se $-$ Se vectors $u = \frac{1}{2} - 2x$, $v = \frac{1}{2}$, $w = 0$ (at 0.343, $\frac{1}{2}$, 0), and $u = \frac{1}{2} - 2x$, $v = \frac{1}{2} - 2y$, $w = \frac{1}{2}$ (at 0.345, $\frac{1}{2}$, $\frac{1}{2}$), were 0.078 and 0.0, respectively. With the y coordinate equal to zero, the Se $-$ Se vectors $u = \frac{1}{2}$, $v = \frac{1}{2}$, $w = \frac{1}{2} - 2z$, and $u = \frac{1}{2}$, $v = \frac{1}{2} - 2y$, $w = 2z$, both appear at ($\frac{1}{2}$, $\frac{1}{2}$, w). There were two such peaks of equal height, located at $w = 0.152$ and $w = 0.335$. From these vectors, the z coordinate is 0.171 or 0.079. The Se $-$ Se vectors $u = 2x$, $v = 2y$, $w = 2z$, and $u = 2x$, $v = 0$, $w = \frac{1}{2} - 2z$, both appear at $v = 0$, giving the same two possibilities in the choice of z coordinate. In the isomorphous salt, $Rb_2Te(S_2O_3)_2 \cdot 1\frac{1}{2}H_2O$,[7] the tellurium coordinates were found to be $x = 0.0715$, $y = 0.0048$, $z = 0.1505$. The first structure factor calculations were therefore based on selenium in the position $x = 0.078$, $y = 0.0$, $z = 0.171$. The selenium contributions gave the probable signs of 250 l even reflections. The electron density map, based on these reflections, had false symmetry due to the omission of l odd reflections. From this Fourier map and the known structure of the rubidium salt, the four sulphur atoms and the two nitrogen atoms could be located. The contributions from these atoms in addition to the selenium contributions gave the probable signs of 782 reflections, including 271 l odd reflections. The oxygen atoms were placed on the basis of the resulting electron density map.

Least squares refinement was then carried out with a full-matrix program, minimizing the function

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

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 3.0 and 0.5, respectively. Unobserved reflections were included with $|F_o|$ equal to the observable limit, when $|F_c|$ exceeded this limit.

With isotropic temperature parameters for all atoms, the reliability index, $R = \sum ||F_o| - |F_c||/\sum|F_o|$, was 0.160. Introducing anisotropic temperature parameters for the selenium and sulphur atoms, the R index dropped to 0.112. At this stage, the calculated structure factors for most reflections of high intensity were much larger than the observed ones. The observed structure factors were therefore corrected for secondary extinction, whereby the reliability index dropped to 0.085. The secondary extinction corrections were carried out by means of a computer program, using the method of Zachariasen.[8]

Table 1. Atomic coordinates, in fractions of cell edges, for ammonium selenopentathionate hemitrihydrate. Origin at a centre of symmetry. The standard deviations from least squares are given in parentheses.

	x	y	z
S(1)	0.0518(1)	0.2471(3)	−0.0218(3)
S(2)	0.0929(1)	0.0437(3)	0.0111(3)
Se	0.0770(1)	0.0148(2)	0.1788(2)
S(4)	0.1563(2)	0.1287(3)	0.2553(3)
S(5)	0.2292(1)	−0.0302(3)	0.2538(3)
O(1)	0.0004(5)	0.2700(9)	0.0556(8)
O(2)	0.1052(5)	0.3470(11)	−0.0131(9)
O(3)	0.0276(5)	0.2288(11)	−0.1280(9)
O(4)	0.1993(5)	−0.1655(9)	0.2744(8)
O(5)	0.2590(4)	−0.0187(9)	0.1487(8)
O(6)	0.2719(4)	0.0173(9)	0.3391(8)
H$_2$O(1)	0.1242(4)	0.3531(1)	0.4977(8)
H$_2$O(2)	0	0.4041(13)	1/4
N(1)	0.2556(4)	0.2564(10)	0.5021(9)
N(2)	0.3944(5)	0.1075(11)	0.2244(10)

Table 2. Temperature parameters in Å^2. For Se 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 and N, 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}
S(1)	320(12)	437(15)	392(24)	−39(11)	44(16)	−75(14)
S(2)	365(13)	448(15)	434(27)	32(12)	−64(18)	−24(15)
Se	301(5)	676(8)	608(14)	−10(6)	238(9)	65(7)
S(4)	557(17)	431(14)	283(22)	157(13)	−40(17)	−24(16)
S(5)	377(12)	283(11)	282(21)	−9(10)	31(15)	−12(12)

	U		U		U
O(1)	532(22)	O(4)	500(22)	H$_2$O(1)	511(23)
O(2)	658(28)	O(5)	521(23)	H$_2$O(2)	556(33)
O(3)	670(28)	O(6)	460(21)	N(1)	388(22)
				N(2)	499(27)

Table 3. Observed and calculated structure factors for ammonium selenopentathionate 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)
2	0	0	81	81
4	0	0	38	33
6	0	0	389	-386
8	0	0	40	20
10	0	0	14	6
12	0	0	164	182
14	0	0	75	85
16	0	0	36	-31
18	0	0	-13	-11
20	0	0	27	-20
22	0	0	30	22
24	0	0	23	18
26	0	0	46	47
1	1	0	226	271
3	1	0	71	-73
5	1	0	134	-147
7	1	0	266	-261
9	1	0	113	-114
11	1	0	94	104
13	1	0	195	199
15	1	0	-12	12
17	1	0	83	-88
19	1	0	84	-80
21	1	0	32	-27
23	1	0	17	12
25	1	0	42	36
0	2	0	187	218
2	2	0	126	-144
4	2	0	96	-90
6	2	0	392	-367
8	2	0	44	-41
10	2	0	79	75
12	2	0	187	192
14	2	0	113	117
16	2	0	70	-74
18	2	0	84	-78
20	2	0	129	-117
22	2	0	17	14
24	2	0	-12	1
26	2	0	52	54
1	3	0	229	241
3	3	0	65	59
5	3	0	290	-285
7	3	0	150	-168
9	3	0	87	79
11	3	0	105	107
13	3	0	102	101
15	3	0	28	28
17	3	0	-13	-12
19	3	0	84	-81
21	3	0	28	-23
23	3	0	32	30
25	3	0	21	20
0	4	0	244	278
2	4	0	70	82
4	4	0	215	221
6	4	0	267	-284
8	4	0	82	-84
10	4	0	52	-49
12	4	0	83	90
14	4	0	66	61
16	4	0	32	25
18	4	0	37	-31
20	4	0	62	-51
22	4	0	49	39
24	4	0	15	14
1	5	0	118	127
3	5	0	40	-38
5	5	0	91	-91
7	5	0	141	-144
9	5	0	-11	7
11	5	0	61	59
13	5	0	49	46
15	5	0	32	28
17	5	0	20	-11
19	5	0	69	-55
21	5	0	29	-27
23	5	0	12	10
0	6	0	91	82
2	6	0	79	-80
4	6	0	56	-53
6	6	0	93	-92
8	6	0	30	-30
10	6	0	47	40
12	6	0	58	56
14	6	0	54	52
16	6	0	25	-19
18	6	0	51	-39
20	6	0	79	-70
22	6	0	20	-17
1	7	0	45	42
3	7	0	-12	-9
5	7	0	20	10
7	7	0	25	-20
9	7	0	56	-48
11	7	0	39	36
13	7	0	77	70
15	7	0	33	25
17	7	0	19	-16
19	7	0	18	-10
21	7	0	24	-22
0	8	0	132	128
2	8	0	26	20
4	8	0	-13	4
6	8	0	27	-23
8	8	0	28	-19
10	8	0	71	-63
12	8	0	36	23
14	8	0	38	25
16	8	0	13	10
18	8	0	12	10
1	9	0	22	12
3	9	0	-14	-1
5	9	0	34	-20
7	9	0	-14	4
9	9	0	54	-42
11	9	0	20	-16
13	9	0	24	16
15	9	0	22	18
17	9	0	-8	-7
0	10	0	55	-44
2	10	0	-14	1
4	10	0	-14	-2
6	10	0	-14	9
8	10	0	-13	2
10	10	0	12	10
12	10	0	13	8
14	10	0	27	21
1	11	0	19	-14
3	11	0	22	17
5	11	0	-11	-4
7	11	0	-10	5
9	11	0	-8	-4
1	1	1	5	-1
2	1	1	116	-120
3	1	1	14	23
4	1	1	149	-156
5	1	1	10	18
6	1	1	138	-136
7	1	1	96	-99
8	1	1	-6	-3
9	1	1	54	55
10	1	1	-7	6
11	1	1	30	-28
12	1	1	12	12
13	1	1	80	-78
14	1	1	-9	5
15	1	1	11	-7
16	1	1	46	43
17	1	1	-10	5
18	1	1	24	25
19	1	1	13	-11
20	1	1	-11	4
21	1	1	11	9
22	1	1	37	-31
23	1	1	-10	-3
24	1	1	20	-20
25	1	1	16	-13
26	1	1	11	-15
0	2	1	68	-69
1	2	1	49	-45
2	2	1	28	-30
3	2	1	76	-74
4	2	1	228	194
5	2	1	8	0
6	2	1	-6	-4
7	2	1	23	20
8	2	1	17	18
9	2	1	17	15
10	2	1	50	54
11	2	1	-8	-7
12	2	1	55	-55
13	2	1	-9	-1
14	2	1	11	-8
15	2	1	11	-11
16	2	1	38	39
17	2	1	16	-12
18	2	1	-10	-2
19	2	1	15	9
20	2	1	-11	-5
21	2	1	18	11
22	2	1	27	24
23	2	1	-10	0
24	2	1	-9	-5
25	2	1	12	-10
26	2	1	-5	-7
1	3	1	82	-92
2	3	1	-5	0
3	3	1	25	-22
4	3	1	27	32
5	3	1	10	12
6	3	1	87	89
7	3	1	34	-38
8	3	1	113	112
9	3	1	92	97
10	3	1	52	45
11	3	1	29	-27
12	3	1	48	-46
13	3	1	34	-32
14	3	1	76	-74
15	3	1	32	-31
16	3	1	45	-42
17	3	1	40	37
18	3	1	-11	7
19	3	1	11	0
20	3	1	21	18
21	3	1	21	15
22	3	1	18	18
23	3	1	-10	2
24	3	1	-8	6
25	3	1	15	-15
0	4	1	49	-46
1	4	1	19	-17
2	4	1	68	-72
3	4	1	102	-100
4	4	1	62	60
5	4	1	-7	-5
6	4	1	21	19
7	4	1	53	52
8	4	1	72	68
9	4	1	71	79
10	4	1	40	-40
11	4	1	-9	1
12	4	1	-9	-5
13	4	1	48	-43
14	4	1	26	-24
15	4	1	32	-32
16	4	1	-10	-10
17	4	1	13	14
18	4	1	39	39
19	4	1	19	22
20	4	1	-11	0
21	4	1	-11	8
22	4	1	20	18
23	4	1	-9	-8
24	4	1	15	-13
25	4	1	-5	-7
1	5	1	20	-17
2	5	1	105	-115
3	5	1	43	-44
4	5	1	97	-102
5	5	1	66	68
6	5	1	41	-45
7	5	1	48	44
8	5	1	18	21
9	5	1	-9	4
10	5	1	-9	7
11	5	1	83	-82
12	5	1	31	27
13	5	1	26	24
14	5	1	-10	-6
15	5	1	68	-68
16	5	1	17	18
17	5	1	27	25
18	5	1	-11	10
19	5	1	25	20
20	5	1	24	19
21	5	1	10	11
22	5	1	-9	-11
23	5	1	10	-6
24	5	1	19	-21
0	6	1	59	67
1	6	1	26	-25
2	6	1	81	-81
3	6	1	72	-77
4	6	1	24	24
5	6	1	30	-31
6	6	1	44	47
7	6	1	25	20
8	6	1	63	62
9	6	1	59	52
10	6	1	48	-47
11	6	1	12	9
12	6	1	12	-9
13	6	1	32	-34
14	6	1	22	-21
15	6	1	29	-27
16	6	1	37	-35
17	6	1	11	5
18	6	1	43	37
19	6	1	24	20
20	6	1	20	17
21	6	1	-8	3
22	6	1	-7	6
1	7	1	21	-26
2	7	1	32	-30
3	7	1	27	32
4	7	1	-10	1
5	7	1	83	88
6	7	1	38	42
7	7	1	20	24
8	7	1	66	69
9	7	1	38	42
10	7	1	21	22
11	7	1	55	-55
12	7	1	31	-28
13	7	1	22	23
14	7	1	57	-53
15	7	1	30	-27
16	7	1	34	-28
17	7	1	20	15
18	7	1	-10	4
19	7	1	12	12
20	7	1	12	14
21	7	1	20	20
0	8	1	-10	-2
1	8	1	12	-8
2	8	1	73	-74
3	8	1	37	-40
4	8	1	35	35
5	8	1	-10	7
6	8	1	-10	-10
7	8	1	13	16
8	8	1	39	38
9	8	1	33	32
10	8	1	-11	-8
11	8	1	-11	1
12	8	1	37	-34
13	8	1	-11	-7
14	8	1	22	-16
15	8	1	25	-19
16	8	1	-10	-7
17	8	1	-9	-6
18	8	1	15	15
19	8	1	8	8
1	9	1	28	-26
2	9	1	38	-37
3	9	1	-11	9
4	9	1	42	-43
5	9	1	37	39
6	9	1	25	-30
7	9	1	15	-17
8	9	1	21	16
9	9	1	50	49
10	9	1	15	13
11	9	1	44	42
12	9	1	13	13
13	9	1	-10	-8
14	9	1	-9	-6
15	9	1	17	-17
16	9	1	10	11
17	9	1	24	24
0	10	1	-21	-17
1	10	1	-11	-7
2	10	1	25	-23
3	10	1	27	-24
4	10	1	48	47
5	10	1	11	-10
6	10	1	15	-14
7	10	1	-10	4
8	10	1	34	28
9	10	1	12	11
10	10	1	-9	0
11	10	1	-9	5
12	10	1	18	-18
13	10	1	-7	-3
14	10	1	15	-15
1	11	1	17	-16
2	11	1	-9	3
3	11	1	-9	-3
4	11	1	9	9
5	11	1	19	18
6	11	1	11	11
7	11	1	-8	-5
8	11	1	10	12
9	11	1	26	28
10	11	1	-5	2
1	0	2	58	-55
2	0	2	267	252
3	0	2	157	-152
4	0	2	54	-62
5	0	2	70	-67
6	0	2	44	-31
7	0	2	131	123
8	0	2	77	68
9	0	2	146	143
10	0	2	28	24
11	0	2	133	131
12	0	2	94	-99
13	0	2	34	-30
14	0	2	137	-130
15	0	2	61	-62
16	0	2	54	50
17	0	2	90	-87
18	0	2	32	28
19	0	2	-12	-3
20	0	2	105	98
21	0	2	35	31
22	0	2	49	41
23	0	2	58	51
24	0	2	14	12
25	0	2	12	13
26	0	2	66	-74
1	1	2	93	-102
2	1	2	150	-148
3	1	2	128	123
4	1	2	165	-159
5	1	2	33	-32
6	1	2	39	-39
7	1	2	73	76
8	1	2	121	113
9	1	2	52	52
10	1	2	133	120
11	1	2	71	64
12	1	2	60	56
13	1	2	115	-112
14	1	2	49	-50
15	1	2	38	-36
16	1	2	81	-81
17	1	2	15	-13
18	1	2	47	-42
19	1	2	64	58
20	1	2	17	13
21	1	2	57	51
22	1	2	58	50
23	1	2	21	16
24	1	2	27	25
25	1	2	44	-39
26	1	2	-6	3
0	2	2	284	-370
1	2	2	93	-106
2	2	2	11	-19
3	2	2	238	-210
4	2	2	205	-175
5	2	2	158	-155
6	2	2	182	170
7	2	2	-7	4
8	2	2	16	-8
9	2	2	82	82
10	2	2	111	110
11	2	2	85	87
12	2	2	38	-37
13	2	2	22	-25
14	2	2	74	-69
15	2	2	49	-67
16	2	2	60	-56
17	2	2	71	-72
18	2	2	39	-32
19	2	2	-12	1
20	2	2	43	41
21	2	2	24	20
22	2	2	14	-11
23	2	2	45	41
24	2	2	-10	-2
25	2	2	-8	7
26	2	2	2	7
1	3	2	46	44
2	3	2	126	-138
3	3	2	124	-122
4	3	2	113	-116
5	3	2	12	-9
6	3	2	62	-58
7	3	2	146	134
8	3	2	78	78
9	3	2	22	20
10	3	2	89	89
11	3	2	28	25
12	3	2	37	37
13	3	2	88	-84
14	3	2	42	-40
15	3	2	28	-27
16	3	2	74	-72
17	3	2	30	-27
18	3	2	36	-31
19	3	2	76	68
20	3	2	12	9
21	3	2	28	27
22	3	2	41	39
23	3	2	-10	-3
24	3	2	25	24
25	3	2	11	-10
0	4	2	55	57
1	4	2	44	-49
2	4	2	52	55
3	4	2	77	-80
4	4	2	14	6
5	4	2	34	-35
6	4	2	144	138
7	4	2	50	55
8	4	2	15	-13
9	4	2	97	94
10	4	2	53	-51
11	4	2	87	83
12	4	2	-10	-8
13	4	2	13	-15

Table 3. Continued.

H	K	L	F(O)	F(C)
14	4	2	47	-44
15	4	2	51	-49
16	4	2	-11	-4
17	4	2	64	-64
18	4	2	45	38
19	4	2	-12	0
20	4	2	121	107
21	4	2	23	20
22	4	2	-10	0
23	4	2	39	39
24	4	2	-8	4
1	5	2	62	-68
2	5	2	70	-77
3	5	2	35	-39
4	5	2	60	-69
5	5	2	-9	11
6	5	2	-9	-4
7	5	2	72	78
8	5	2	64	66
9	5	2	52	-49
10	5	2	68	70
11	5	2	31	-37
12	5	2	36	33
13	5	2	34	-33
14	5	2	27	-28
15	5	2	18	-18
16	5	2	42	-43
17	5	2	14	-14
18	5	2	25	-22
19	5	2	40	33
20	5	2	-11	6
21	5	2	-10	13
22	5	2	28	27
23	5	2	-8	-8
0	6	2	112	-135
1	6	2	23	-26
2	6	2	11	8
3	6	2	68	-80
4	6	2	70	-71
5	6	2	57	-62
6	6	2	60	67
7	6	2	-10	-1
8	6	2	66	69
9	6	2	30	33
10	6	2	41	46
11	6	2	46	51
12	6	2	26	-27
13	6	2	-11	-2
14	6	2	27	-22
15	6	2	24	-22
16	6	2	-12	-8
17	6	2	35	-34
18	6	2	-12	-5
19	6	2	-11	-2
20	6	2	-10	0
21	6	2	11	11
22	6	2	11	-0
1	7	2	67	-76
2	7	2	35	-43
3	7	2	50	54
4	7	2	47	-52
5	7	2	36	41
6	7	2	17	-19
7	7	2	20	23
8	7	2	22	24
9	7	2	14	11
10	7	2	27	29
11	7	2	16	-13
12	7	2	16	14
13	7	2	33	-32
14	7	2	17	-16
15	7	2	-12	10
16	7	2	26	-26
17	7	2	-11	10
18	7	2	15	-16
19	7	2	-9	2
20	7	2	-8	3
21	7	2	-6	8
0	8	2	25	-29
1	8	2	11	-10
2	8	2	14	13
3	8	2	25	-23
4	8	2	50	55
5	8	2	14	-12
6	8	2	-11	1
7	8	2	16	17
8	8	2	-12	-4
9	8	2	28	29
10	8	2	17	-20
11	8	2	19	20
12	8	2	49	-50
13	8	2	-12	-6
14	8	2	18	-19
15	8	2	17	-16
16	8	2	41	43
17	8	2	19	-23
18	8	2	13	15
19	8	2	-7	-1
1	9	2	23	-22
2	9	2	17	-12
3	9	2	-12	0
4	9	2	12	-11
5	9	2	24	24
6	9	2	-12	-6
7	9	2	-12	-6
8	9	2	19	17

H	K	L	F(O)	F(C)
9	9	2	17	12
10	9	2	18	19
11	9	2	20	-20
12	9	2	11	10
13	9	2	29	-25
14	9	2	-10	-4
15	9	2	-9	3
16	9	2	15	-14
17	9	2	20	24
0	10	2	56	-50
1	10	2	-12	-8
2	10	2	46	-37
3	10	2	20	-18
4	10	2	20	18
5	10	2	16	-13
6	10	2	40	35
7	10	2	-11	-1
8	10	2	-11	1
9	10	2	14	9
10	10	2	-10	-4
11	10	2	11	10
12	10	2	-8	-1
13	10	2	-7	2
1	11	2	-9	-1
2	11	2	-9	-7
3	11	2	13	-13
4	11	2	11	-8
5	11	2	26	25
6	11	2	-8	-6
7	11	2	22	20
8	11	2	-7	1
9	11	2	-6	2
1	1	3	50	59
2	1	3	52	-58
3	1	3	-2	-3
4	1	3	86	-94
5	1	3	24	-30
6	1	3	23	-22
7	1	3	43	38
8	1	3	18	14
9	1	3	102	-111
10	1	3	28	27
11	1	3	42	-40
12	1	3	22	22
13	1	3	11	16
14	1	3	11	15
15	1	3	20	-20
16	1	3	58	60
17	1	3	7	-10
18	1	3	34	35
19	1	3	15	17
20	1	3	-5	-3
21	1	3	11	-8
22	1	3	43	-36
23	1	3	5	-4
24	1	3	27	-26
25	1	3	-4	0
0	2	3	61	55
1	2	3	23	-17
2	2	3	32	33
3	2	3	19	17
4	2	3	123	-117
5	2	3	29	24
6	2	3	44	50
7	2	3	31	27
8	2	3	6	1
9	2	3	43	-41
10	2	3	61	-63
11	2	3	8	6
12	2	3	44	41
13	2	3	-4	-2
14	2	3	-4	3
15	2	3	-5	4
16	2	3	29	-27
17	2	3	6	10
18	2	3	-5	4
19	2	3	8	10
20	2	3	11	10
21	2	3	7	5
22	2	3	26	-24
23	2	3	10	-13
24	2	3	6	5
25	2	3	17	-15
1	3	3	14	13
2	3	3	14	13
3	3	3	5	-6
4	3	3	29	31
5	3	3	29	27
6	3	3	82	89
7	3	3	94	98
8	3	3	25	21
9	3	3	26	-26
10	3	3	7	-10
11	3	3	55	60
12	3	3	92	-96
13	3	3	47	49
14	3	3	55	-52
15	3	3	42	41
16	3	3	19	-19
17	3	3	23	-24
18	3	3	37	35
19	3	3	9	9
20	3	3	7	7
21	3	3	15	-13
22	3	3	-5	2
23	3	3	-8	0

H	K	L	F(O)	F(C)
24	3	3	-4	-5
25	3	3	17	18
0	4	3	30	-37
1	4	3	30	-37
2	4	3	38	-37
3	4	3	7	7
4	4	3	13	13
5	4	3	65	-67
6	4	3	8	-6
7	4	3	11	-13
8	4	3	32	29
9	4	3	45	-48
10	4	3	19	16
11	4	3	44	-42
12	4	3	15	-12
13	4	3	46	-46
14	4	3	-5	1
15	4	3	-5	-3
16	4	3	15	16
17	4	3	36	37
18	4	3	30	-32
19	4	3	35	32
20	4	3	7	8
21	4	3	9	-6
22	4	3	24	-21
23	4	3	25	-25
24	4	3	10	10
1	5	3	16	4
2	5	3	33	-33
3	5	3	55	55
4	5	3	23	-23
5	5	3	33	-37
6	5	3	-4	2
7	5	3	42	-41
8	5	3	17	-13
9	5	3	40	-40
10	5	3	21	-11
11	5	3	13	-14
12	5	3	56	-62
13	5	3	41	38
14	5	3	52	53
15	5	3	35	36
16	5	3	17	-15
17	5	3	39	39
18	5	3	-5	-5
19	5	3	13	9
20	5	3	7	-8
21	5	3	27	-28
22	5	3	7	2
23	5	3	4	4
0	6	3	62	-60
1	6	3	24	-22
2	6	3	86	88
3	6	3	21	21
4	6	3	14	18
5	6	3	6	-7
6	6	3	7	-2
7	6	3	50	45
8	6	3	28	-27
9	6	3	7	2
10	6	3	35	36
11	6	3	21	-22
12	6	3	7	-4
13	6	3	38	-35
14	6	3	-5	3
15	6	3	14	12
16	6	3	48	44
17	6	3	41	35
18	6	3	27	-26
19	6	3	30	25
20	6	3	-4	-3
21	6	3	13	-10
22	6	3	7	-7
0	7	3	33	31
1	7	3	21	-20
2	7	3	31	33
3	7	3	33	34
4	7	3	27	25
5	7	3	51	-50
6	7	3	59	58
7	7	3	19	16
8	7	3	6	5
9	7	3	12	-10
10	7	3	55	56
11	7	3	35	-41
12	7	3	27	-26
13	7	3	27	26
14	7	3	25	-23
15	7	3	27	27
16	7	3	-5	-7
17	7	3	11	-11
18	7	3	26	25
19	7	3	6	-6
20	7	3	-3	-6
0	8	3	46	-46
1	8	3	-5	-6
2	8	3	37	33
3	8	3	17	14
4	8	3	34	-36
5	8	3	14	12
6	8	3	23	21
7	8	3	18	17
8	8	3	27	-27
9	8	3	27	-23
10	8	3	7	-6
11	8	3	26	-25
12	8	3	22	19

H	K	L	F(O)	F(C)
13	8	3	22	-19
14	8	3	-5	0
15	8	3	-5	6
16	8	3	10	11
17	8	3	20	20
18	8	3	-13	-13
1	9	3	20	20
2	9	3	-5	0
3	9	3	8	-5
4	9	3	14	-12
5	9	3	24	-24
6	9	3	-5	-5
7	9	3	22	20
8	9	3	19	-18
9	9	3	19	18
10	9	3	15	-10
11	9	3	11	-9
12	9	3	15	13
13	9	3	14	12
14	9	3	27	22
15	9	3	16	14
16	9	3	11	-9
0	10	3	16	14
1	10	3	11	-9
2	10	3	25	26
3	10	3	21	20
4	10	3	32	-30
5	10	3	-5	4
6	10	3	32	30
7	10	3	-5	6
8	10	3	18	-14
9	10	3	15	-13
10	10	3	-4	-4
11	10	3	7	-5
12	10	3	11	13
1	11	3	-4	0
2	11	3	18	16
3	11	3	-4	-2
4	11	3	25	22
5	10	3	-5	4
6	11	3	19	21
7	11	3	17	19
8	11	3	7	-8
2	0	4	52	-50
3	0	4	246	259
4	0	4	130	-120
5	0	4	138	141
6	0	4	60	-49
7	0	4	43	-40
8	0	4	53	54
9	0	4	108	-111
10	0	4	16	12
11	0	4	120	-120
12	0	4	7	4
13	0	4	12	-11
14	0	4	15	-26
15	0	4	27	32
16	0	4	40	-45
17	0	4	62	73
18	0	4	64	61
19	0	4	18	20
20	0	4	64	68
21	0	4	20	-23
22	0	4	46	43
23	0	4	44	-44
24	0	4	6	-8
25	0	4	8	-9
1	1	4	37	10
2	1	4	88	93
3	1	4	122	-128
4	1	4	142	145
5	1	4	88	87
6	1	4	66	62
7	1	4	83	-81
8	1	4	77	-78
9	1	4	51	49
10	1	4	122	-122
11	1	4	51	-55
12	1	4	76	-75
13	1	4	85	85
14	1	4	37	40
15	1	4	98	-101
16	1	4	87	89
17	1	4	11	-11
18	1	4	56	60
19	1	4	-5	1
20	1	4	11	-12
21	1	4	46	47
22	1	4	65	-57
23	1	4	-5	0
24	1	4	33	-34
25	1	4	-3	-7
0	2	4	55	-56
1	2	4	46	49
2	2	4	20	8
3	2	4	114	115
4	2	4	41	-26
5	2	4	56	55
6	2	4	33	30
7	2	4	40	-39
8	2	4	56	56
9	2	4	110	-114
10	2	4	99	98
11	2	4	81	-83
12	2	4	13	15
13	2	4	23	24

H	K	L	F(O)	F(C)
14	2	4	6	-5
15	2	4	71	77
16	2	4	83	-85
17	2	4	91	97
18	2	4	9	-8
19	2	4	10	9
20	2	4	29	-23
21	2	4	36	-34
22	2	4	31	32
23	2	4	67	-65
24	2	4	22	-23
25	2	4	21	-23
1	3	4	48	48
2	3	4	133	141
3	3	4	5	4
4	3	4	93	95
5	3	4	39	-40
6	3	4	39	39
7	3	4	50	-45
8	3	4	87	-87
9	3	4	170	162
10	3	4	94	-93
11	3	4	38	-40
12	3	4	38	-39
13	3	4	-5	-2
14	3	4	27	33
15	3	4	71	-72
16	3	4	68	73
17	3	4	30	33
18	3	4	36	37
19	3	4	22	-21
20	3	4	-6	-7
21	3	4	38	35
22	3	4	43	-41
23	3	4	12	14
24	3	4	32	-34
0	4	4	80	-79
1	4	4	9	-8
2	4	4	6	-3
3	4	4	119	120
4	4	4	106	101
5	4	4	89	86
6	4	4	82	-79
7	4	4	24	22
8	4	4	26	-27
9	4	4	74	-77
10	4	4	19	-21
11	4	4	89	-95
12	4	4	38	-32
13	4	4	20	-23
14	4	4	24	-24
15	4	4	32	34
16	4	4	12	12
17	4	4	56	56
18	4	4	28	22
19	4	4	6	4
20	4	4	20	17
21	4	4	15	-13
22	4	4	49	49
23	4	4	29	-32
1	5	4	25	-16
2	5	4	79	85
3	5	4	8	-8
4	5	4	78	84
5	5	4	38	33
6	5	4	17	13
7	5	4	41	-44
8	5	4	47	-51
9	5	4	86	80
10	5	4	64	-65
11	5	4	22	-21
12	5	4	31	-34
13	5	4	-5	-4
14	5	4	27	29
15	5	4	28	-28
16	5	4	46	43
17	5	4	34	34
18	5	4	27	25
19	5	4	-5	-3
20	5	4	-5	-4
21	5	4	23	22
22	5	4	24	-26
0	6	4	39	-39
1	6	4	6	10
2	6	4	54	-45
3	6	4	59	59
4	6	4	24	-26
5	6	4	17	18
6	6	4	52	51
7	6	4	33	-32
8	6	4	26	22
9	6	4	46	-45
10	6	4	40	40
11	6	4	39	-41
12	6	4	21	-19
13	6	4	19	21
14	6	4	9	-7
15	6	4	43	43
16	6	4	27	-25
17	6	4	44	43
18	6	4	-5	0
19	6	4	-5	3
20	6	4	20	-21
21	6	4	22	-21
1	7	4	16	-14
2	7	4	30	31

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	7	4	21	-19	9	2	5	48	48	20	6	5	10	-11	10	1	6	50	57	21	5	6	22	-25
4	7	4	45	44	10	2	5	44	41	21	6	5	8	11	11	1	6	176	174	0	6	6	33	34
5	7	4	72	72	11	2	5	11	-8	1	7	5	15	11	12	1	6	-11	1	1	6	6	-11	8
6	7	4	9	9	12	2	5	24	-28	2	7	5	55	-59	13	1	6	16	24	2	6	6	24	2
7	7	4	33	28	13	2	5	-10	-3	3	7	5	31	-28	14	1	6	32	-36	3	6	6	40	-42
8	7	4	31	-32	14	2	5	21	23	4	7	5	23	-25	15	1	6	69	75	4	6	6	46	-44
9	7	4	9	-11	15	2	5	13	-10	5	7	5	45	47	16	1	6	36	-35	5	6	6	26	-24
10	7	4	34	-35	16	2	5	35	40	6	7	5	19	20	17	1	6	61	-65	6	6	6	38	-39
11	7	4	26	-23	17	2	5	21	-18	7	7	5	-11	5	18	1	6	-13	-11	7	6	6	17	-14
12	7	4	16	-14	18	2	5	-12	0	8	7	5	63	64	19	1	6	34	-34	8	6	6	-12	1
13	7	4	16	18	19	2	5	-12	7	9	7	5	31	30	20	1	6	24	23	9	6	6	-13	12
14	7	4	21	20	20	2	5	22	-22	10	7	5	29	33	21	1	6	27	-25	10	6	6	37	38
15	7	4	24	-23	21	2	5	16	11	11	7	5	18	-19	22	1	6	24	22	11	6	6	28	30
16	7	4	31	28	22	2	5	18	18	12	7	5	19	-18	23	1	6	19	24	12	6	6	41	43
17	7	4	-5	1	23	2	5	-9	4	13	7	5	54	53	24	1	6	-8	5	13	6	6	16	19
18	7	4	15	15	24	2	5	-8	-2	14	7	5	45	-48	0	2	6	102	88	14	6	6	48	43
19	7	4	13	11	1	3	5	19	-15	15	7	5	-11	-7	1	2	6	43	-43	15	6	6	-13	2
20	7	4	8	-9	2	3	5	28	-22	16	7	5	36	-36	2	2	6	89	82	16	6	6	-13	8
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8	10	4	14	11	20	4	5	30	-32	6	10	5	34	-31	21	3	6	36	-36	2	9	6	16	11
9	10	4	20	-16	21	4	5	14	16	7	10	5	13	16	22	3	6	-10	9	3	9	6	16	14
10	10	4	6	8	22	4	5	-9	4	8	10	5	-9	4	23	3	6	-8	6	4	9	6	16	14
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12	10	4	11	-10	1	5	5	26	18	10	10	5	-8	5	1	4	6	13	-16	6	9	6	-13	-9
1	11	4	24	-23	2	5	5	102	-102	11	10	5	11	13	2	4	6	52	50	7	9	6	23	-21
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3	11	4	12	15	4	5	5	92	-94	1	11	5	-8	-6	4	4	6	45	-36	9	9	6	-12	0
4	11	4	8	6	5	5	5	9	9	2	11	5	-7	-1	5	4	6	28	20	10	9	6	-11	-3
5	11	4	10	8	6	5	5	12	-10	3	11	5	-7	-2	6	4	6	70	-73	11	9	6	-11	7
6	11	4	-3	-1	7	5	5	33	-29	4	11	5	-7	-3	7	4	6	-10	10	12	9	6	-10	-4
7	11	4	11	13	8	5	5	41	43	5	11	5	-6	5	8	4	6	84	-90	13	9	6	-9	4
1	1	5	38	-33	9	5	5	50	-51	6	11	5	-6	4	9	4	6	35	37	14	9	6	-7	3
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5	1	5	57	-59	13	5	5	47	47	4	0	6	75	-81	13	4	6	32	-32	3	10	6	-11	-6
6	1	5	58	-73	14	5	5	18	-20	5	0	6	18	-14	14	4	6	38	38	4	10	6	-11	11
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17	1	5	27	25	2	6	5	51	-46	16	0	6	37	36	4	5	6	34	-36	2	1	7	37	-38
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22	1	5	22	-19	7	6	5	33	32	21	0	6	37	38	9	5	6	78	-78	7	1	7	18	16
23	1	5	-10	-1	8	6	5	13	10	22	0	6	-12	2	10	5	6	17	17	8	1	7	19	-16
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25	1	5	9	-12	10	6	5	33	-35	24	0	6	31	37	12	5	6	39	43	10	1	7	-10	-8
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6	2	5	36	-31	17	6	5	11	-8	7	1	6	86	-91	19	5	6	18	-16	17	1	7	16	13
7	2	5	47	42	18	6	5	31	30	8	1	6	47	49	20	5	6	-10	8	18	1	7	21	21
8	2	5	-8	-5	19	6	5	12	13	9	1	6	65	-65						19	1	7	43	41

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)
20	1	7	-13	3	23	3	7	-6	-8	5	6	7	-12	1	1	9	7	-13	-6	3	0	10	76	77
21	1	7	11	10	0	4	7	96	-89	6	6	7	40	42	2	9	7	-13	-7	4	0	10	75	-77
22	1	7	18	-18	1	4	7	-9	3	7	6	7	12	11	3	9	7	-12	-5	5	0	10	76	67
23	1	7	-8	-2	2	4	7	-9	0	8	6	7	-12	6	4	9	7	-12	-7	6	0	10	57	-61
0	2	7	10	-19	3	4	7	23	-19	9	6	7	20	18	5	9	7	-12	6	7	0	10	54	50
1	2	7	6	12	4	4	7	28	-18	10	6	7	20	20	6	9	7	-12	-10	8	0	10	47	-38
2	2	7	12	-16	5	4	7	20	20	11	6	7	23	-25	7	9	7	36	37	9	0	10	26	-26
3	2	7	29	-22	6	4	7	55	55	12	6	7	37	-40	8	9	7	-11	-5	10	0	10	50	51
4	2	7	63	-53	7	4	7	-10	3	13	6	7	24	-24	9	9	7	41	-40	11	0	10	61	-55
5	2	7	26	27	8	4	7	-11	6	14	6	7	28	-26	10	9	7	-10	-1	12	0	10	-22	-12
6	2	7	80	80	9	4	7	32	33	15	6	7	13	4	11	9	7	-9	-7	13	0	10	44	-43
7	2	7	-9	-4	10	4	7	-11	-1	16	6	7	36	38	0	10	7	22	-21	14	0	10	32	-33
8	2	7	-9	5	11	4	7	14	-21	17	6	7	11	14	1	10	7	-10	3	15	0	10	27	12
9	2	7	25	-27	12	4	7	40	-42	18	6	7	-10	2	2	10	7	-10	8	16	0	10	-21	10
10	2	7	48	-53	13	4	7	15	-15	19	6	7	13	16	4	10	7	-10	-5	17	0	10	39	25
11	2	7	-11	10	14	4	7	27	-29	20	6	7	12	20	6	10	7	17	-20	18	0	10	49	-41
12	2	7	-11	5	15	4	7	-13	-4	1	7	7	65	-68	7	10	7	-8	-9	19	0	10	-23	15
13	2	7	-12	-3	16	4	7	-13	11	2	7	7	12	17	0	0	2	129	-125	20	0	10	-14	11
14	2	7	-12	-5	17	4	7	18	16	3	7	7	15	16	0	0	4	330	426	1	0	12	41	-37
15	2	7	-12	-7	18	4	7	-13	-8	4	7	7	-12	7	0	0	6	159	179	2	0	12	48	-42
16	2	7	16	-14	19	4	7	24	22	5	7	7	-13	10	0	0	8	136	153	3	0	12	27	-14
17	2	7	-13	5	20	4	7	20	22	6	7	7	22	25	0	0	10	45	-43	4	0	12	-22	-10
18	2	7	-13	12	21	4	7	-9	-8	7	7	7	59	57	0	0	12	117	128	5	0	12	-22	6
19	2	7	-13	6	22	4	7	11	-13	8	7	7	18	18	0	0	14	63	-53	6	0	12	67	-50
20	2	7	22	21	1	5	7	47	-49	9	7	7	-13	12	0	0	16	35	45	7	0	12	32	25
21	2	7	-11	4	2	5	7	39	-34	10	7	7	-13	0	1	0	8	53	-43	8	0	12	-23	14
22	2	7	12	-18	3	5	7	46	44	11	7	7	24	24	2	0	8	97	-105	9	0	12	53	46
23	2	7	-8	-4	4	5	7	20	-18	12	7	7	16	-19	3	0	8	21	13	10	0	12	27	-23
1	3	7	34	-34	5	5	7	-11	11	13	7	7	50	-49	4	0	8	25	15	11	0	12	27	18
2	3	7	10	-11	6	5	7	20	-22	14	7	7	14	-17	5	0	8	-18	4	12	0	12	50	43
3	3	7	15	-15	7	5	7	-11	12	15	7	7	-11	6	6	0	8	33	26	13	0	12	29	-24
4	3	7	24	24	8	5	7	-11	7	16	7	7	-10	-1	7	0	8	36	28	14	0	12	27	20
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6	3	7	63	56	10	5	7	21	-21	18	7	7	-7	5	9	0	8	42	50	16	0	12	27	-25
7	3	7	125	118	11	5	7	20	-25	0	8	7	73	-72	10	0	8	61	-53	17	0	12	23	-20
8	3	7	44	40	12	5	7	-13	-9	1	8	7	16	15	11	0	8	34	37	1	0	14	-21	23
9	3	7	13	9	13	5	7	73	-79	2	8	7	16	10	12	0	8	46	-42	2	0	14	-21	5
10	3	7	13	8	14	5	7	13	11	3	8	7	24	-22	13	0	8	34	-32	3	0	14	-21	-9
11	3	7	25	25	15	5	7	28	33	4	8	7	13	-11	14	0	8	77	-74	4	0	14	-21	-2
12	3	7	29	-32	16	5	7	18	19	5	8	7	19	21	15	0	8	56	-59	5	0	14	-20	7
13	3	7	-12	0	17	5	7	12	15	6	8	7	63	62	16	0	8	51	-52	6	0	14	-20	6
14	3	7	32	-32	18	5	7	-11	15	7	8	7	-13	6	17	0	8	44	-45	7	0	14	27	22
15	3	7	-13	3	19	5	7	23	27	8	8	7	-13	4	18	0	8	50	50	8	0	14	35	24
16	3	7	-13	-4	20	5	7	11	13	9	8	7	-13	5	19	0	8	-21	10	9	0	14	-18	17
17	3	7	24	-23	21	5	7	11	13	10	8	7	-12	-6	20	0	8	59	50	10	0	14	26	29
18	3	7	18	19	0	6	7	114	-117	11	8	7	-12	-11	21	0	8	21	16	11	0	14	21	-11
19	3	7	12	12	1	6	7	-11	12	12	8	7	-11	-14	22	0	8	23	25	12	0	14	21	-23
20	3	7	-12	0	2	6	7	35	32	13	8	7	-10	-6	1	0	10	54	57	13	0	14	27	-25
21	3	7	-10	-10	3	6	7	28	-28	14	8	7	21	-23	2	0	10	76	76	0	0	16	35	45
22	3	7	-9	-7	4	6	7	14	17	15	8	7	-8	4						1	0	16	-11	-16
										16	8	7	-6	8						2	0	16	22	-15

The formula used is $F_{corr} = KF_o(1 + \beta CI_o)$, where $\beta = 2(1 + \cos^4 2\theta)/(1 + \cos^2 2\theta)^2$, and C was found to be 1.03×10^{-6}. In the last refinement cycles, all parameters (83) except the scale factors were refined simultaneously. No calculated shift of a parameter exceeded one fifth of its standard deviation. The final value of the R index, including non-observed reflections with $|F_o|$ equal to the observable limit when $|F_c|$ exceeds this limit, was 0.085.

The final atomic parameters, with standard deviations from least squares, are listed in Table 1 and 2. The observed structure factors, and the calculated ones based on the parameters of Table 1 and 2, are listed in Table 3.

A three-dimensional difference electron density map, based on the data listed in Table 3, was calculated. The map showed no peaks higher than 0.7 e/Å³. All the hydrogen atoms, placed in accordance with the assumed hydrogen bonds listed in Table 5, could be located in regions of the map with electron density of about 0.3 e/Å³. No attempt was made, however, to include the hydrogen atoms in the structure factor calculations.

THE SELENOPENTATHIONATE ION

The selenopentathionate ion contains a non-planar S − S − Se − S − S chain, with three oxygen atoms bonded to each of the terminal sulphur atoms. The S − S − Se − S − S chain occurs in the *trans* form, and conforms approximately

to twofold axis symmetry, the axis passing through the selenium atom. The dimensions of the ion, calculated from the atomic coordinates of Table 1, are listed in Table 4. The standard deviations in unit cell dimensions are small

Table 4. Dimensions of the selenopentathionate ion. Standard deviations are given in parentheses.

Bond lengths and angles

S(1) − S(2) = 2.120(4) Å   S(4) − S(5) = 2.116(4) Å
S(2) − Se = 2.153(4)   Se − S(4) = 2.181(3)
∠S(1) − S(2) − Se = 104.0(2)°   ∠Se − S(4) − S(5) = 101.1(2)°
                 ∠S(2) − Se − S(4) = 104.9(2)°

S(1) − O(1) = 1.459(10) Å   S(5) − O(4) = 1.430(9) Å
S(1) − O(2) = 1.450(11)   S(5) − O(5) = 1.464(10)
S(1) − O(3) = 1.438(12)   S(5) − O(6) = 1.460(10)
∠S(2) − S(1) − O(1) = 107.0(4)°   ∠S(4) − S(5) − O(4) = 107.9(4)°
∠S(2) − S(1) − O(2) = 104.7(4)°   ∠S(4) − S(5) − O(5) = 104.9(4)°
∠S(2) − S(1) − O(3) = 102.4(4)°   ∠S(4) − S(5) − O(6) = 102.3(4)°
∠O(1) − S(1) − O(2) = 114.3(6)°   ∠O(4) − S(5) − O(5) = 114.2(5)°
∠O(1) − S(1) − O(3) = 112.6(6)°   ∠O(4) − S(5) − O(6) = 113.4(5)°
∠O(2) − S(1) − O(3) = 114.4(6)°   ∠O(5) − S(5) − O(6) = 112.8(5)°

Dihedral angles

S(1)S(2)Se/S(2)SeS(4) = 83.3°   S(2)SeS(4)/SeS(4)S(5) = 85.2°

SeS(2)S(1)/S(2)S(1)O(1) = 28.0°   SeS(4)S(5)/S(4)S(5)O(4) = 38.0°
SeS(2)S(1)/S(2)S(1)O(2) = 93.7°   SeS(4)S(5)/S(4)S(5)O(5) = 84.1°
SeS(2)S(1)/S(2)S(1)O(3) = 146.6°   SeS(4)S(5)/S(4)S(5)O(6) = 157.9°
S(2)S(1)O(1)/S(2)S(1)O(2) = 121.7°   S(4)S(5)O(4)/S(4)S(5)O(5) = 122.1°
S(2)S(1)O(1)/S(2)S(1)O(3) = 118.6°   S(4)S(5)O(4)/S(4)S(5)O(6) = 119.9°
S(2)S(1)O(2)/S(2)S(1)O(3) = 119.7°   S(4)S(5)O(5)/S(4)S(5)O(6) = 118.0°

Non-bonded distances

S(1) − Se = 3.368(4) Å   Se − S(5) = 3.319(3) Å
S(1) − S(4) = 4.252(5)   S(2) − S(5) = 4.218(5)
S(2) − S(4) = 3.437(5)   S(1) − S(5) = 5.678(4)

and have been neglected. Fig. 1 gives a view of the selenopentathionate ion, as seen along the approximate twofold axis, with principal bond lengths and angles, and also a view normal to this axis and to a line through S(2) and the coordinate midpoint of the four sulphur atoms.

The selenium-sulphur bonds, between a divalent selenium atom and a divalent sulphur atom, are 2.153(4) Å and 2.181(3) Å, and thus slightly, but significantly different in the two halves of the chain. The corresponding bond lengths found in the crystals of barium selenopentathionate dihydrate [4] are 2.180(3) Å, and in the crystals of barium selenopentathionate trihydrate,[5] 2.181(2) Å and 2.178(2) Å. The bonds are all a little shorter than the sum of the single covalent bond radii for selenium and sulphur, 2.21 Å.[10]

The lengths of the two terminal sulphur-sulphur bonds, 2.120(4) Å and 2.116(4) Å, agree with the corresponding lengths in the isomorphous salts,
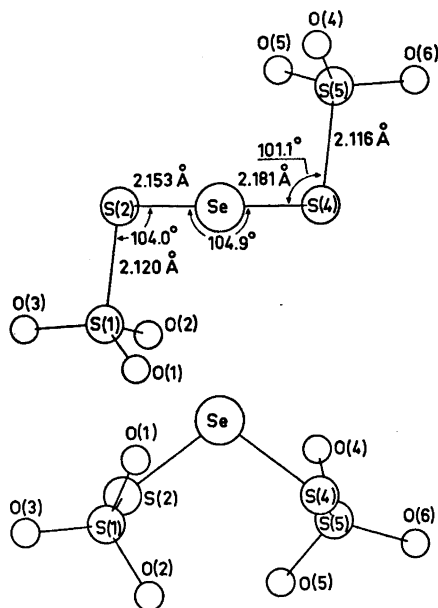
Fig. 1. The *trans* from of the selenopenta-
thionate ion in $(NH_4)_2Se(S_2O_3)_2.1\frac{1}{2}H_2O$, as
seen along an approximate twofold axis
(above), and normal to this axis and to a
line through S(2) and the coordinate mid-
point of the four sulphur atoms (below).

rubidium telluropentathionate hemitrihydrate, 2.116(11) Å and 2.126(9) Å,[2,7]
and potassium pentathionate hemitrihydrate, 2.124(6) Å and 2.110(6) Å.[2]
The weighted mean of these six values is 2.118(3) Å, with an average devia-
tion of 0.005 Å. In the barium salts of the selenopentathionate ion, these
sulphur-sulphur bonds are 2.096(3) Å,[4] and 2.104(2) Å and 2.093(2) Å.[5]
The $S-Se-S$ bond angle is 104.9(2)° in the present salt, compared to 103.2(2)°
and 104.0(1)° in the barium salts.[4,5]

The $Se-S-S$ angles, 104.0(2)° and 101.1(2)°, and the SSeS/SeSS dihedral
angles, 83.3° and 85.2°, are slightly different in the two halves of the ion.
The average of the dihedral angles in the present salt is 22° smaller than the
average of the corresponding angles in the two barium selenopentathionate
hydrates,[4,5] where the $S-S-Se-S-S$ chain has the *cis* form.

The three oxygen atoms and the two sulphur atoms of each half of the
selenopentathionate ion form a thiosulphate group with distorted tetrahedral
shape. The $S-S-O$ angles and $O-S-O$ angles range from 102.3(4)° to
107.9(4)°, and from 112.6(6)° to 114.4(6)°, respectively. The $S-O$ bond lengths
range from 1.430(9) Å to 1.464(10) Å; the average length is 1.450(4) Å. As
seen from the data in Table 4, the bond lengths and bond angles in the two
thiosulphate groups are within the errors the same. The SSO/SSO dihedral
angles are also approximately the same in the two thiosulphate groups,
whereas the SeSS/SSO dihedral angles differ by 9.6°, 10.0°, and 11.3°. This
corresponds to a difference in rotation about the $S-S$ bonds of about 10.3°.

A *trans* selenopentathionate ion, with the two halves of the ion exactly
equal, would possess the symmetry of a twofold axis passing through the
selenium atom. The deviations from twofold axis symmetry in the present

salt, as evidenced from the slightly different Se−S bonds, Se−S−S bond angles, SSeS/SeSS dihedral angles, and degrees of rotation about the S−S bonds in the two halves of the ion, are probably due to crystal packing effects and to hydrogen bonding between the ammonium ions, the water molecules, and the thiosulphate oxygen atoms.

Table 5. Distances (Å), and angles (°) between directions, from N(1) and N(2) to oxygen atoms (above), and from $H_2O(1)$ and $H_2O(2)$ to oxygen and nitrogen atoms (below), indicating hydrogen bonds. Standard deviations of the distances and angles are 0.011 − 0.016 Å and 0.4°, respectively.

$N(1A) - H_2O(1A) = 2.867$ $N(2) - H_2O(1A) = 2.903$
$N(1A) - O(4B)\ \ \ = 3.103$ $N(2) - H_2O(2D) = 2.914$
$N(1A) - O(5)\ \ \ \ \ = 3.079$ $N(2) - O(3E)\ \ \ \ = 2.898$
$N(1A) - O(5C)\ \ \ = 2.885$ $N(2) - O(6)\ \ \ \ \ \ = 3.038$
$\angle\ H_2O(1A) - N(1A) - O(4B) = 101.2$ $\angle\ H_2O(1A) - N(2) - H_2O(2D) = 106.9$
$\angle\ H_2O(1A) - N(1A) - O(5)\ = 70.7$ $\angle\ H_2O(1A) - N(2) - O(3E)\ \ \ = 129.6$
$\angle\ H_2O(1A) - N(1A) - O(5C) = 106.1$ $\angle\ H_2O(1A) - N(2) - O(6)\ \ \ \ \ = 113.1$
$\angle\ O(4B) - N(1A) - O(5)\ \ \ \ = 113.4$ $\angle\ H_2O(2D) - N(2) - O(3E)\ \ \ = 81.6$
$\angle\ O(4B) - N(1A) - O(5C) = 139.8$ $\angle\ H_2O(2D) - N(2) - O(6)\ \ \ \ \ = 113.2$
$\angle\ O(5) - N(1A) - O(5C)\ \ \ \ = 103.3$ $\angle\ O(3E) - N(2) - O(6)\ \ \ \ \ \ \ = 107.8$

$H_2O(1A) - O(1F) = 2.828$ $H_2O(2D) - O(1D) = 2.749$
$H_2O(1A) - O(2D) = 2.779$ $H_2O(2D) - O(1G) = 2.749$
$H_2O(1A) - N(1A) = 2.867$ $H_2O(2D) - N(2)\ \ \ = 2.914$
$H_2O(1A) - N(2)\ \ \ = 2.903$ $H_2O(2D) - N(2H) = 2.914$
$\angle\ O(1F) - H_2O(1A) - O(2D) = 97.8$ $\angle\ O(1D) - H_2O(2D) - O(1G)\ \ = 125.9$
$\angle\ O(1F) - H_2O(1A) - N(1A) = 143.0$ $\angle\ O(1D) - H_2O(2D) - N(2)\ \ \ = 113.4$
$\angle\ O(1F) - H_2O(1A) - N(2)\ \ = 98.6$ $\angle\ O(1D) - H_2O(2D) - N(2H) = 101.2$
$\angle\ O(2D) - H_2O(1A) - N(1A) = 116.4$ $\angle\ O(1G) - H_2O(2D) - N(2)\ \ \ = 101.2$
$\angle\ O(2D) - H_2O(1A) - N(2)\ \ = 84.4$ $\angle\ O(1G) - H_2O(2D) - N(2H) = 113.4$
$\angle\ N(1A) - H_2O(1A) - N(2)\ \ = 98.4$ $\angle\ N(2) - H_2O(2D) - N(2H)\ \ = 98.8$
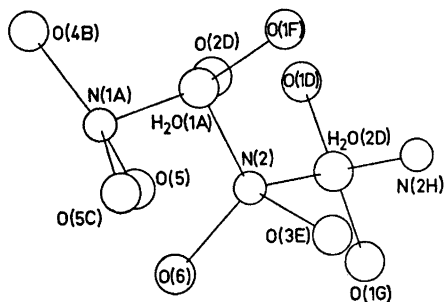
Fig. 2. N−H···O and O−H···O hydrogen bonding in $(NH_4)_2Se(S_2O_3)_2 \cdot 1\frac{1}{2}H_2O$, as seen along the *b* axis.

The closest interionic Se−Se approach is Se−Se′ = 3.661(2) Å, where Se′ is at $\bar{x},y,\frac{1}{2}-z$ relative to Se. The angles between the directions from Se to S(2), S(4), and Se′ are, $\angle S(2) - Se - S(4) = 105°$, $\angle S(4) - Se - Se' = 116°$, and $\angle Se' - Se - S(2) = 128°$. Se′ is located 1.80 Å from the plane through Se, S(2), and S(4). There is no interionic Se−S distance shorter than 4.12 Å, and no interionic S−S distance shorter than 3.83 Å.

HYDROGEN BONDING

Like the rubidium ions in rubidium telluropentathionate hemitrihydrate,[7] each of the ammonium ions in the present structure is surrounded by seven oxygen atoms. The distances are in the range $2.87 - 3.23$ Å. Four of these approaches are probably hydrogen bonds of the type $N - H \cdots O$. In each case, the arrangement around nitrogen is a distorted tetrahedral one, with distances and angles as listed in Table 5. $N(1)$ is bonded to one water oxygen atom and to three oxygen atoms of three different selenopentathionate ions. $N(2)$ is bonded to two water oxygen atoms and to two oxygen atoms of different selenopentathionate ions.

Each of the water oxygen atoms accepts two $N - H \cdots O$ hydrogen bonds from two different ammonium ions. In addition, each water molecule appears to form hydrogen bonds to two thiosulphate oxygen atoms. The arrangement of nitrogen atoms and thiosulphate oxygen atoms around the water oxygen atoms is distorted tetrahedral. The data are given in Table 5. A view of the $N - H \cdots O$ hydrogen bonds, as seen along the b axis, is shown in Fig. 2. In Table 5 and Fig. 2, the letter A denotes an atom at $\frac{1}{2} - x, \frac{1}{2} - y, -\frac{1}{2} + z$, B at $x, \bar{y}, -\frac{1}{2} + z$, C at $\frac{1}{2} - x, \frac{1}{2} + y, z$, D at $\frac{1}{2} - x, -\frac{1}{2} + y, z$, E at $\frac{1}{2} - x, \frac{1}{2} - y, \frac{1}{2} + z$, F at $\frac{1}{2} + x, \frac{1}{2} - y, \bar{z}$, G at $\frac{1}{2} + x, -\frac{1}{2} + y, \frac{1}{2} - z$, and H at $1 - x, y, \frac{1}{2} - z$, relative to the coordinates of Table 1. The lengths of the $N - H \cdots O$ bonds, $2.867 - 3.103$ Å, and of the $O - H \cdots O$ bonds, $2.749 - 2.828$ Å, are in the range found for this type of hydrogen bonds in other compounds.[11]

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