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The Crystal Structure of Potassium Pentathionate Hemitrihydrate

KJARTAN MARØY

Chemical Institute, University of Bergen, Bergen, Norway

The salt, $K_2S(S_2O_3)_2.1\frac{1}{2}H_2O$, crystallizes in the orthorhombic space group *Pbcn* (No. 60) with eight formula units per unit cell, and $a = 20.316(10)$ Å, $b = 9.229(4)$ Å, $c = 12.248(7)$ Å. The structure has been determined by X-ray methods, and refined by least squares for the $hk0-hk4$ and $h0l-h4l$ reflections.

The pentathionate ion has the *trans* form in this salt, the sulphonate groups being located on opposite sides of the plane through the three middle, divalent sulphur atoms. The dimensions of the five-membered sulphur chain, from one end of the chain to the other, are: S(1) – S(2) = 2.124(6) Å, S(2) – S(3) = 2.021(7) Å, S(3) – S(4) = 2.036(7) Å, S(4) – S(5) = 2.110(6) Å, ∠S(1) – S(2) – S(3) = 103.1(3)°, ∠S(2) – S(3) – S(4) = 107.4(3)°, ∠S(3) – S(4) – S(5) = 103.1(3)°. The dihedral angles, S(1)S(2)S(3)/S(2)S(3)S(4) and S(2)S(3)S(4)/S(3)S(4)S(5), are 81.9° and 84.4°, respectively.

The crystal structures of three salts of pentathionic acid have been reported earlier, by Foss and co-workers. The three salts are barium salts: one orthorhombic [1] and one triclinic [2] dihydrate, and one acetone solvate.[3] The five-membered, non-planar sulphur chain of the pentathionate ion has the *cis* rotational-isomeric form in these salts, the terminal atoms being located on the same side of the plane through the three middle atoms.

In the potassium salt, $K_2S(S_2O_3)_2.1\frac{1}{2}H_2O$, the pentathionate ion has now been found to occur in the *trans* form; the terminal sulphur atoms are located on opposite sides of the plane through the three middle atoms.

A series of isomorphous alkali (and ammonium) telluropentathionate, selenopentathionate, and pentathionate hemitrihydrates were prepared in 1950 by Foss and Jahr.[4] The crystal structures of three representatives of this series, including one telluropentathionate, $Rb_2Te(S_2O_3)_2.1\frac{1}{2}H_2O$,[5] one selenopentathionate, $(NH_4)_2Se(S_2O_3)_2.1\frac{1}{2}H_2O$,[6] and one pentathionate, $K_2S(S_2O_3)_2.1\frac{1}{2}H_2O$ (present work), have been determined. Some features of the structures have been briefly reported earlier.[7]

Debus [8] in 1888 established the composition of potassium pentathionate, crystallized from water or dilute acids, as that of a hemitrihydrate. The unit cell and space group were reported by Foss and Jahr.[4]

EXPERIMENTAL

Potassium pentathionate hemitrihydrate, $K_2S(S_2O_3).1\frac{1}{2}H_2O$, was prepared from sodium thiosulphate as described by Stamm *et al.*,[9] and recrystallized from dilute hydrochloric acid.

The salt forms colourless 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. 10, p. 122). The unit cell is orthorhombic, with axial lengths $a = 20.316(10)$ Å, $b = 9.229(4)$ Å, $c = 12.248(7)$ 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.[4]

Using the multiple-film technique, integrated zero-layer and equi-inclination Weissenberg photographs were taken with Cu$K\alpha$ radiation (Ni-filtered) for the $hk0 - hk4$ and $h0l - h4l$ reflections. The intensities were estimated visually. Altogether, 1005 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 lower as well as the upper parts of the films. At high angles, 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.

The intensities were corrected for absorption ($\mu = 156$ cm^{-1}) by the method described by Coppens *et al.*[11] 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 6, 10, and 10 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 ($\bar{1}00$)	0.0111 cm	0.0104 cm
(110) and ($\bar{1}\bar{1}0$)	0.0098	0.0142
($1\bar{1}0$) and ($\bar{1}10$)	0.0093	0.0164
(001) and ($00\bar{1}$)	0.0194	0.0186

The scattering curves, for K^+, S, and O, used for the calculations of structure factors, were those given in *International Tables for X-ray Crystallography*,[10] Table 3.3.1A.

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

THE STRUCTURE ANALYSIS

The first structure factor calculations were based on the two potassium atoms and the five sulphur atoms, with coordinates derived from the coordinates of the corresponding atoms in the isomorphous salts, $Rb_2Te(S_2O_3)_2.1\frac{1}{2}H_2O$,[5] and $(NH_4)_2Se(S_2O_3)_2.1\frac{1}{2}H_2O$,[6] assuming bond lengths and angles of the pentathionate ion as found in the barium salts.[1–3] Three-dimensional Fourier summations were made of 356 reflections, with signs based on the potassium and sulphur contributions, and the resulting map allowed the location of the oxygen atoms. The next structure factor calculation, including all atoms with an overall isotropic thermal parameter, gave a reliability index, $R=\sum||F_o|-|F_c||\sum|F_o|$, of 0.245.

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 0.7, respectively. Unobserved reflections were included with $|F_o|$ equal to the observable limit when $|F_c|$ exceeded this limit. Using individual isotropic temperature parameters, the R index was reduced to 0.135.

The observed structure factors were then corrected for secondary extinction according to Zachariasen.[12] 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 2.70×10^{-7}. The absorption term in β was omitted.

After additional refinement cycles, with anisotropic temperature parameters for the potassium and sulphur atoms, no calculated shift of a parameter exceeded 0.3 times its standard deviation. All parameters (93), not including scale factors, were refined simultaneously in the last cycles. The final value of the R index, with non-observed reflections included when $|F_c|$ exceeds the observable limit, was 0.093.

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, in fraction of cell edges, for potassium pentathionate hemitrihydrate. Origin at a centre of symmetry. Standard deviations from least squares are given in parentheses.

	x	y	z
K(1)	0.2619(2)	0.2481(5)	0.4965(4)
K(2)	0.3944(2)	0.1224(6)	0.2165(4)
S(1)	0.0514(2)	0.2610(5)	−0.0190(4)
S(2)	0.0914(2)	0.0520(5)	0.0101(4)
S(3)	0.0774(2)	0.0242(6)	0.1721(4)
S(4)	0.1522(2)	0.1280(5)	0.2506(4)
S(5)	0.2286(2)	−0.0269(4)	0.2496(4)
O(1)	−0.0013(6)	0.2801(13)	0.0600(10)
O(2)	0.1054(6)	0.3583(15)	−0.0083(11)
O(3)	0.0272(7)	0.2442(15)	−0.1260(11)
O(4)	0.1981(6)	−0.1644(13)	0.2685(10)
O(5)	0.2595(5)	−0.0109(14)	0.1429(9)
O(6)	0.2694(5)	0.0195(13)	0.3359(10)
$H_2O(1)$	0.1311(5)	0.3379(13)	0.4957(9)
$H_2O(2)$	0	0.4300(20)	1/4

Table 3. Temperature parameters in Å^2. For K 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}
K(1)	607(24)	640(26)	779(28)	50(23)	92(30)	70(24)
K(2)	646(25)	959(39)	813(35)	157(28)	78(34)	−132(25)
S(1)	327(17)	559(29)	605(29)	3(21)	64(33)	−61(20)
S(2)	439(20)	462(25)	690(32)	−7(20)	−52(30)	−56(23)
S(3)	394(19)	736(39)	750(34)	−19(24)	328(34)	129(24)
S(4)	721(28)	532(29)	505(27)	222(26)	−27(32)	4(26)
S(5)	483(18)	292(21)	433(22)	−18(18)	77(28)	−29(20)

	U		U		U
O(1)	679(35)	O(4)	620(33)	$H_2O(1)$	616(33)
O(2)	819(41)	O(5)	668(34)	$H_2O(2)$	775(55)
O(3)	860(41)	O(6)	599(33)		

A three-dimensional difference electron density map, based on the data of Table 4, showed no peaks higher than 1.0 e/Å^{-3}. The map allowed the location of the water hydrogen atoms, but no attempt was made to include these atoms in the structure factor calculations.

THE PENTATHIONATE ION

The dimensions of the pentathionate ion, calculated from the atomic coordinates of Table 2, are listed in Table 5, with standard deviations from least squares in parentheses. The uncertainties in unit cell dimensions are small and have been neglected.

The bond lengths and angles are within the errors the same in the two halves of the sulphur chain. The non-bonded distances S(1) − S(4) and S(2) − S(5) differ by 0.037 Å. This is due to differences in the degrees of rotation about the middle S − S bonds; the dihedral angles, S(1)S(2)S(3)/S(2)S(3)S(4) and S(2)S(3)S(4)/S(3)S(4)S(5), are 81.9° and 84,4°, respectively. The five-membered sulphur chain has the *trans* form in this salt, and apart from the small differences in the dihedral angles, the chain possesses, within the errors, the symmetry of a twofold axis passing through the middle sulphur atom. Fig. 1 gives a view of the pentathionate 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 S(1), S(2), S(4), and S(5).

The bond lengths and angles of the *trans* sulphur chain in the present salt do not deviate significantly from the bond lengths and angles of the rotational-isomeric *cis* form of the ion, found in the barium salts. The mean values of the terminal S − S bond lengths, of the middle S − S bond lengths, of the therminal S − S − S angles, and of the middle S − S − S angles in the two dimorphs of barium pentathionate dihydrate and the solvate of barium pentathionate with acetone, are 2.12 Å, 2.04 Å, 105°, and 106°, respectively, with estimated

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

H	K	L	F(O)	F(C)
2	0	0	42	-55
4	0	0	66	76
6	0	0	387	-368
8	0	0	181	181
10	0	0	13	-12
12	0	0	103	102
14	0	0	-12	-5
16	0	0	40	-36
18	0	0	24	21
20	0	0	33	31
22	0	0	43	42
24	0	0	13	-11
1	1	0	87	92
3	1	0	86	-79
5	1	0	29	-24
7	1	0	153	-151
9	1	0	91	-93
11	1	0	34	32
13	1	0	122	124
15	1	0	-13	2
17	1	0	62	-69
19	1	0	46	-45
21	1	0	-15	-1
23	1	0	19	17
25	1	0	12	11
0	2	0	35	19
2	2	0	148	-129
4	2	0	78	-96
6	2	0	202	-197
8	2	0	-9	-5
10	2	0	90	95
12	2	0	96	97
14	2	0	59	61
16	2	0	65	-69
18	2	0	38	-36
20	2	0	64	-66
22	2	0	-15	10
24	2	0	-12	-3
1	3	0	209	202
3	3	0	45	39
5	3	0	236	-215
7	3	0	93	-92
9	3	0	108	102
11	3	0	48	50
13	3	0	22	22
15	3	0	25	-19
17	3	0	15	9
19	3	0	17	-19
21	3	0	-15	4
23	3	0	16	20
25	3	0	-8	-4
0	4	0	167	186
2	4	0	50	-44
4	4	0	334	343
6	4	0	190	-189
8	4	0	14	-10
10	4	0	100	-102
12	4	0	50	46
14	4	0	23	15
16	4	0	27	28
18	4	0	-15	5
20	4	0	27	-28
22	4	0	40	38
24	4	0	10	8
1	5	0	21	16
3	5	0	-11	-10
5	5	0	45	-45
7	5	0	77	-74
9	5	0	48	49
11	5	0	36	39
13	5	0	-14	-7
15	5	0	17	-16
17	5	0	-15	4
19	5	0	-15	-15
21	5	0	20	-22
23	5	0	-9	-2
0	6	0	20	-7
2	6	0	80	-78
4	6	0	46	-41
6	6	0	-13	1
8	6	0	-14	-7
10	6	0	44	33
12	6	0	-15	9
14	6	0	27	29
16	6	0	25	-17
18	6	0	-15	-11
20	6	0	27	-33
22	6	0	10	-10
1	7	0	-14	13
3	7	0	-14	8
5	7	0	71	67
7	7	0	14	15
9	7	0	46	-44
11	7	0	21	-26
13	7	0	35	28
15	7	0	20	20
17	7	0	-14	11
19	7	0	-11	10
21	7	0	-7	-5
0	8	0	91	95
2	8	0	28	-33
4	8	0	22	27
6	8	0	26	-19
8	8	0	23	22
10	8	0	57	-62
12	8	0	-15	-5
14	8	0	15	-5
16	8	0	-13	3
18	8	0	22	18
1	9	0	21	-27
3	9	0	-15	-12
5	9	0	-15	12
7	9	0	33	35
9	9	0	-15	-12
11	9	0	23	-22
13	9	0	-13	8
15	9	0	12	15
0	10	0	62	-53
2	10	0	-14	-2
4	10	0	-14	-1
6	10	0	26	25
8	10	0	-13	6
10	10	0	-12	4
12	10	0	11	-12
1	1	1	38	48
2	1	1	110	-120
3	1	1	-7	2
4	1	1	133	-126
5	1	1	21	-24
6	1	1	172	-175
7	1	1	106	-104
8	1	1	38	39
9	1	1	82	79
10	1	1	25	-28
11	1	1	-14	6
12	1	1	46	50
13	1	1	79	-79
14	1	1	21	-21
15	1	1	28	-27
16	1	1	69	69
17	1	1	16	14
18	1	1	-19	3
19	1	1	-20	4
20	1	1	18	19
21	1	1	-20	5
22	1	1	36	-33
23	1	1	-18	-6
24	1	1	-16	-9
25	1	1	-13	-8
0	2	1	131	-149
1	2	1	48	-45
2	2	1	42	-47
3	2	1	79	-77
4	2	1	252	232
5	2	1	36	27
6	2	1	36	37
7	2	1	35	33
8	2	1	46	-38
9	2	1	25	26
10	2	1	22	18
11	2	1	-15	-7
12	2	1	33	-29
13	2	1	-16	-12
14	2	1	18	16
15	2	1	30	-28
16	2	1	35	37
17	2	1	16	-10
18	2	1	-19	-8
19	2	1	15	14
20	2	1	18	-18
21	2	1	18	13
22	2	1	17	16
23	2	1	-18	-2
24	2	1	-15	7
25	2	1	-12	-6
1	3	1	54	-50
2	3	1	12	-5
3	3	1	39	-36
4	3	1	-12	1
5	3	1	21	-23
6	3	1	119	123
7	3	1	49	-51
8	3	1	75	72
9	3	1	100	102
10	3	1	74	74
11	3	1	-16	-2
12	3	1	73	-73
13	3	1	31	-33
14	3	1	42	-42
15	3	1	36	-38
16	3	1	67	-67
17	3	1	36	37
18	3	1	22	19
19	3	1	-20	8
20	3	1	-20	4
21	3	1	-20	3
22	3	1	27	29
23	3	1	-16	-4
24	3	1	-14	0
0	4	1	22	-18
1	4	1	29	-25
2	4	1	31	-36
3	4	1	104	-99
4	4	1	53	49
5	4	1	55	-49
6	4	1	-14	7
7	4	1	34	33
8	4	1	47	41
9	4	1	69	68
10	4	1	32	-27
11	4	1	-17	13
12	4	1	-18	5
13	4	1	33	-35
14	4	1	-19	-11
15	4	1	-19	-15
16	4	1	23	-16
17	4	1	16	15
18	4	1	27	28
19	4	1	19	22
20	4	1	-20	0
21	4	1	-19	2
22	4	1	-17	11
23	4	1	-15	-7
24	4	1	-11	-9
1	5	1	30	-27
2	5	1	103	-116
3	5	1	32	-33
4	5	1	75	-79
5	5	1	66	68
6	5	1	53	-56
7	5	1	28	27
8	5	1	37	40
9	5	1	20	-15
10	5	1	-18	-13
11	5	1	85	-86
12	5	1	49	42
13	5	1	42	37
14	5	1	24	-27
15	5	1	34	-37
16	5	1	24	27
17	5	1	-20	15
18	5	1	-20	2
19	5	1	-20	2
20	5	1	34	30
21	5	1	-17	5
22	5	1	-15	-17
23	5	1	-12	-4
0	6	1	97	99
1	6	1	-17	-10
2	6	1	53	-49
3	6	1	60	-56
4	6	1	-17	-8
5	6	1	-17	7
6	6	1	17	19
7	6	1	35	32
8	6	1	67	65
9	6	1	52	49
10	6	1	-19	-14
11	6	1	-19	-6
12	6	1	-19	-4
13	6	1	32	-32
14	6	1	-20	-7
15	6	1	35	-34
16	6	1	26	-27
17	6	1	-20	-4
18	6	1	32	28
19	6	1	-18	11
20	6	1	-16	14
21	6	1	-14	11
22	6	1	-11	5
1	7	1	19	-23
2	7	1	-18	-6
3	7	1	47	-45
4	7	1	-18	-7
5	7	1	66	64
6	7	1	39	39
7	7	1	-19	-1
8	7	1	40	40
9	7	1	24	24
10	7	1	36	37
11	7	1	49	-52
12	7	1	41	-36
13	7	1	23	31
14	7	1	22	-27
15	7	1	-20	-13
16	7	1	35	-31
17	7	1	-18	5
18	7	1	-17	11
19	7	1	-15	-4
20	7	1	-12	1
0	8	1	19	18
1	8	1	-19	-16
2	8	1	52	-51
3	8	1	35	-36
4	8	1	33	31
5	8	1	-19	-13
6	8	1	-20	-13
7	8	1	-20	0
8	8	1	21	25
9	8	1	-20	11
10	8	1	-20	-6
11	8	1	-20	11
12	8	1	30	-27
13	8	1	-20	5
14	8	1	-19	-3
15	8	1	-18	0
16	8	1	-17	-1
17	8	1	-15	-2
18	8	1	-13	11
19	8	1	-9	8
1	9	1	-19	-4
2	9	1	30	-25
3	9	1	-19	4
4	9	1	23	-20
5	9	1	20	25
6	9	1	24	-24
7	9	1	27	-28
8	9	1	20	11
9	9	1	45	45
10	9	1	-19	-6
11	9	1	24	-26
12	9	1	19	16
13	9	1	-17	1
14	9	1	-15	-5
15	9	1	18	-18
16	9	1	16	14
0	10	1	19	-9
1	10	1	-19	9
2	10	1	19	-23
3	10	1	-19	2
4	10	1	40	40
5	10	1	-18	8
6	10	1	-18	-16
7	10	1	-18	1
2	0	2	208	192
3	0	2	93	-97
4	0	2	27	52
5	0	2	-6	-1
6	0	2	110	-106
7	0	2	119	119
8	0	2	19	8
9	0	2	98	89
10	0	2	52	-57
11	0	2	51	51
12	0	2	15	-8
13	0	2	32	-31
14	0	2	57	-58
15	0	2	26	-22
16	0	2	54	51
17	0	2	22	-24
18	0	2	-13	-9
19	0	2	-13	3
20	0	2	48	45
21	0	2	-14	9
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Table 4. Continued.

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10	3	11	-18	13	15	4	5	-17	-15	12	4	7	33	-35	12	4	9	-19	6	3	4	12	-18	-5
11	3	11	-18	14	16	4	5	-17	-8	13	4	7	-18	-16	13	4	9	-18	-16	4	4	12	22	30
12	3	11	20	-15	17	4	5	-17	7	14	4	7	-18	-17	14	4	9	-17	5	5	4	12	23	19
13	3	11	-16	-2	18	4	5	18	20	15	4	7	-18	-6	15	4	9	-16	2	6	4	12	35	-49
14	3	11	-14	-7	19	4	5	16	14	16	4	7	-17	16	16	4	9	-15	0	7	4	12	-17	16
15	3	11	-12	6	20	4	5	15	-16	17	4	7	-17	10	17	4	9	-13	14	8	4	12	-17	4
16	3	11	11	-10	21	4	5	-13	5	18	4	7	-15	-2	18	4	9	-11	14	9	4	12	-16	9
1	3	12	-18	9	0	4	6	141	138	19	4	7	-14	15	0	4	10	-17	-5	10	4	12	-15	8

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

Bond lengths and angles

S(1) – S(2) = 2.124(6) Å
S(2) – S(3) = 2.021(7)
∠S(1) – S(2) – S(3) = 103.1(3)°

S(4) – S(5) = 2.110(6) Å
S(3) – S(4) = 2.036(7)
∠S(3) – S(4) – S(5) = 103.1(3)°

∠S(2) – S(3) – S(4) = 107.4(3)°

S(1) – O(1) = 1.453(13) Å
S(1) – O(2) = 1.424(14)
S(1) – O(3) = 1.408(15)
∠S(2) – S(1) – O(1) = 106.3(5)°
∠S(2) – S(1) – O(2) = 105.2(6)°
∠S(2) – S(1) – O(3) = 100.9(6)°
∠O(1) – S(1) – O(2) = 115.5(8)°
∠O(1) – S(1) – O(3) = 112.1(8)°
∠O(2) – S(1) – O(3) = 115.1(8)°

S(5) – O(4) = 1.431(12) Å
S(5) – O(5) = 1.457(12)
S(5) – O(6) = 1.410(12)
∠S(4) – S(5) – O(4) = 106.4(5)°
∠S(4) – S(5) – O(5) = 104.7(5)°
∠S(4) – S(5) – O(6) = 102.9(5)°
∠O(4) – S(5) – O(5) = 114.9(7)°
∠O(4) – S(5) – O(6) = 113.7(7)°
∠O(5) – S(5) – O(6) = 112.8(7)°

Dihedral angles

S(1)S(2)S(3)/S(2)S(3)S(4) = 81.9°

S(3)S(2)S(1)/S(2)S(1)O(1) = 30.6°
S(3)S(2)S(1)/S(2)S(1)O(2) = 92.4°
S(3)S(2)S(1)/S(2)S(1)O(3) = 147.6°
S(2)S(1)O(1)/S(2)S(1)O(2) = 122.9°
S(2)S(1)O(1)/S(2)S(1)O(3) = 117.1°
S(2)S(1)O(2)/S(2)S(1)O(3) = 120.0°

S(2)S(3)S(4)/S(3)S(4)S(5) = 84.4°

S(3)S(4)S(5)/S(4)S(5)O(4) = 38.9°
S(3)S(4)S(5)/S(4)S(5)O(5) = 83.2°
S(3)S(4)S(5)/S(4)S(5)O(6) = 158.6°
S(4)S(5)O(4)/S(4)S(5)O(5) = 122.1°
S(4)S(5)O(4)/S(4)S(5)O(6) = 119.8°
S(4)S(5)O(5)/S(4)S(5)O(6) = 118.1°

Non-bonded distances

S(1) – S(3) = 3.246(7) Å
S(1) – S(4) = 4.074(7)
S(2) – S(4) = 3.270(7)

S(3) – S(5) = 3.248(5) Å
S(2) – S(5) = 4.111(6)
S(1) – S(5) = 5.553(6)

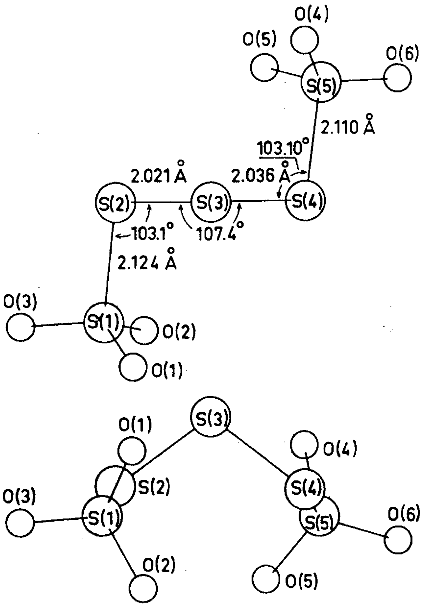

Fig. 1. The *trans* form of the pentathionate ion in $K_2S(S_2O_3)_2.1\frac{1}{2}H_2O$, as seen along an approximate twofold axis (above), and normal to this axis and to a line through S(2) and the coordinate midpoint of S(1), S(2), S(4), and S(5) (below).

standard deviations of 0.02 Å in bond lengths, and 1.5 – 2.0° in bond angles.[3] The mean values of the corresponding bond lengths and angles in the present salt are 2.117(5) Å, 2.029(5) Å, 103.1(2)°, and 107.4(3)°, the latter angle being observed only once. The mean value of the SSS/SSS dihedral angles in the barium salts is 108°, and thus larger by 25° than the mean value of the dihedral angles in the present salt.

The bond lengths and angles are within the errors the same in the two thiosulphate groups formed by the two sulphur atoms and three oxygen atoms of each half of the pentathionate ion. The shape of the groups is distorted tetrahedral, with S – S – O angles in the range 100.9(6)° to 106.4(5)°, and O – S – O angles in the range 112.1(8)° to 115.5(8)°. The S – O bonds range from 1.408(15) Å to 1.457(12) Å, with an average of 1.431(5) Å. The SSO/SSO dihedral angles are approximately the same in the two groups, whereas the SSS/SSO dihedral angles differ by 8.3°, 9.2°, and 11.0°, with an average difference of 9.5°, compared to 10.3° in the crystals of ammonium selenopentathionate hemitrihydrate.[6]

THE IONIC ENVIRONMENT

The arrangement of oxygen atoms around the potassium ions is similar to that of oxygen atoms around the rubidium ions in the isomorphous salt, $Rb_2Te(S_2O_3)_2.1\frac{1}{2}H_2O$.[5] Each of the potassium ions is surrounded by seven oxygen atoms, at distances 2.78 – 3.25 Å for K(1) and 2.78 – 3.17 Å for K(2). This is within the range for K – O distances found in other salts.[13,14] The

Table 6. Distances (Å), and angles (°) between directions, from K(1) to oxygen atoms (above), and from K(2) to oxygen atoms (below). Standard deviations of the distances and angles are 0.011 – 0.013 Å, and 0.3 – 0.4°, respectively.

				I	II	III	IV	V	VI
I	$H_2O(1)$	(x,y,z)	2.78						
II	O(6)	(x,y,z)	2.89	105					
III	O(2)	$(\frac{1}{2}-x,\frac{1}{2}-y,\frac{1}{2}+z)$	2.87	177	72				
IV	O(5)	$(\frac{1}{2}-x,\frac{1}{2}-y,\frac{1}{2}+z)$	3.05	68	172	115			
V	O(4)	$(\frac{1}{2}-x,\frac{1}{2}+y,z)$	3.02	100	63	80	112		
VI	O(6)	$(\frac{1}{2}-x,\frac{1}{2}+y,z)$	3.25	65	99	116	73	44	
VII	O(5)	$(x,\bar{y},\frac{1}{2}+z)$	2.83	102	82	76	104	143	168

				I	II	III	IV	V	VI
I	$H_2O(1)$	$(\frac{1}{2}-x,\frac{1}{2}-y,-\frac{1}{2}+z)$	2.78						
II	$H_2O(2)$	$(\frac{1}{2}-x,-\frac{1}{2}+y,z)$	2.82	112					
III	O(5)	(x,y,z)	3.14	67	117				
IV	O(6)	(x,y,z)	3.08	110	111	45			
V	O(3)	$(\frac{1}{2}-x,\frac{1}{2}-y,\frac{1}{2}+z)$	2.79	136	75	151	106		
VI	O(3)	$(\frac{1}{2}+x,\frac{1}{2}-y,\bar{z})$	3.17	76	69	143	171	65	
VII	O(4)	$(\frac{1}{2}-x,\frac{1}{2}+y,z)$	2.79	90	157	76	64	85	113

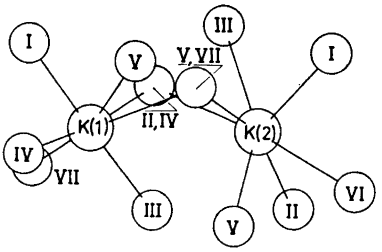

Fig. 2. A view of the potassium-oxygen coordination in $K_2S(S_2O_3)_2.1\frac{1}{2}H_2O$, as seen along the *b* axis. The oxygen atoms are marked I – VII in accordance with Table 6.

individual distances and related angles are listed in Table 6, and a view of the arrangement is shown in Fig. 2. Four of the K(1) – O distances are in the range 2.78 – 2.89 Å, and four of the K(2) – O distances in the range 2.78 – 2.82 Å, whereas the remaining three K(1) – O and three K(2) – O distances are in the ranges 3.02 – 3.25 Å and 3.08 – 3.17 Å, respectively. The sum of the ionic radii for potassium and oxygen is 2.73 Å,[15] and it is difficult to assess whether the approaches of 3.02 – 3.25 Å represent significant contacts or not. Assuming the coordination number to be four or seven, the coordination polyhedron of oxygen atoms around the potassium ions is in either case an irregular one. O(5) and O(6) are each included three times in the potassium oxygen approaches, O(3) and O(4) twice, and O(2) once, whereas O(1) is the only oxygen atom not included.

The water molecule $H_2O(1)$ forms hydrogen bonds to O(1′) and O(2′), at distances $H_2O(1)-O(1')=2.775(16)$ Å, and $H_2O(1)-O(2')=2.853(19)$ Å, and $\angle O(1')-H_2O(1)-O(2')=90.6(5)°$. Here, O(1′) is at $\bar{x},y,\frac{1}{2}-z$ relative to O(1), and O(2′) is at $x,1-y,\frac{1}{2}+z$ relative to O(2). The water molecule $H_2O(2)$, situated on a twofold axis, forms hydrogen bonds to O(1), at $H_2O(2)-O(1)=2.707(16)$ and $\angle O(1)-H_2O(2)-O(1')=118.6(6)°$. The final difference electron density map confirmed the positions of the hydrogen atoms, indicated by these hydrogen bonds. The oxygen atom O(1), not included in the potassium oxygen approaches, is connected to both of the water molecules, at a $H_2O(1)-O(1)-H_2O(2)$ angle of 97.0(4)°.

The closest interionic S–S approach is $S(3)-S(3')=3.697(6)$ Å, where $S(3')$ is at $\bar{x},y,\frac{1}{2}-z$ relative to S(3).

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