460. The Crystal Structures of the Acid Salts of Some Monobasic Acids. Part IX.[1] Rubidium Hydrogen Bisglycollate

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Rubidium hydrogen bisglycollate, $RbH(C_2H_3O_3)_2$, crystallises with four molecules in a cell, belonging to the space group $P2_1/n$, with $a = 4{\cdot}052$, $b = 17{\cdot}91$, $c = 10{\cdot}52$ Å, $\beta = 98° 26'$. The potassium acid salt is isomorphous. The structure of the rubidium salt has been determined and refined by three-dimensional least-squares analysis to $R = 9{\cdot}3\%$. In accordance with the indications of the infrared spectrum, the structure is that of an acid salt of type *B*: the glycollate residues are crystallographically distinguishable as glycollate anion and glycollic acid molecule. These species are joined by a short, though not symmetrical, hydrogen bond with $O \cdots O = 2{\cdot}53$ Å between their carboxyl groups. The resulting bisglycollate units are linked into a three-dimensional array by weaker hydrogen bonds, with $O \cdots O = 2{\cdot}73$ Å, between a hydroxyl groups of one and a carboxyl group of another.

ACID salts of glycollic acid, $CH_2(OH)CO_2H$, have been known for a century. An ammonium salt, $NH_4(C_2H_3O_3)_2$, was described by Heintz,[2] and a hydrated sodium salt, $NaH(C_2H_3O_3)_2,H_2O$, by de Forcrand.[3] An acid potassium salt, $KH(C_2H_3O_3)_2$, is easily prepared, and its infrared spectrum suggested that it was[4] of type *B* with the two anions of the formula non-equivalent. Since all but two of the crystalline acid salts we have studied hitherto have been of type *A*, with the anions equivalent, this bisglycollate was an interesting subject for study.

We also prepared the rubidium analogue. Our intention was to make use of its probable isomorphism to facilitate solution of the phase problem, and then to concentrate the effort of structural refinement on the potassium compound, which would have a lower absorption for *X*-rays. Isomorphism was indeed evident in the 0*kl*-photographs; but higher layer-line photographs showed the crystals of the potassium salt to be twinned in a way that we could not easily understand, so that satisfactory intensity measurements would have been hard to come by. The analysis was therefore completed with $RbH(C_2H_3O_3)_2$.

EXPERIMENTAL

Preparation of Crystals.—Potassium, or rubidium, hydrogen bisglycollate is prepared by dissolving a 1/2 equivalent of potassium hydroxide, or rubidium carbonate, in a little 50%

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[1] Part VIII, H. H. Mills and J. C. Speakman, *J.*, 1963, 4355.
[2] W. Heintz, *Ann. Physik*, 1861, **114**, 451.
[3] R. de Forcrand, *Bull. Soc. chim. France*, 1883, (2) **39**, 309.
[4] H. N. Shrivastava and J. C. Speakman, *J.*, 1961, 1151.

aqueous ethanol, adding 1 equivalent of glycollic acid, in the form of the " 67% solution " commercially available, and allowing the solution to stand, or to evaporate. The crystals that appear are prismatic, elongated in the *a*-direction. They can be recrystallised from aqueous ethanol, in which they are very soluble. Their compositions were checked roughly by titrimetric determination of the equivalent, and confirmed by structure analysis.

Crystal Data.—The following values were established by photographic methods with CuK_α-radiation ($\lambda = 1{\cdot}5418$ Å):

Potassium Hydrogen Bisglycollate, $KH(C_2H_3O_3)_2$, $M = 190{\cdot}2$ *Monoclinic,* $a = 3{\cdot}97$, $b = 17{\cdot}62$, $c = 10{\cdot}40$ Å, $\beta = 100° \, 24'$, $U = 716$ Å^3, $D_m = 1{\cdot}7$, $Z = 4$, $D_c = 1{\cdot}76$. Isomorphous with rubidium salt.

Rubidium Hydrogen Bisglycollate, $RbH(C_2H_3O_3)_2$, $M = 236{\cdot}6$ *Monoclinic,* $a = 4{\cdot}052 \pm 0{\cdot}004$, $b = 17{\cdot}91 \pm 0{\cdot}01$, $c = 10{\cdot}52 \pm 0{\cdot}01$ Å, $\beta = 98° \, 26' \pm 4'$, $U = 755$ Å^3, $D_m = 2{\cdot}05$, $Z = 4$, $D_c = 2{\cdot}08$, μ(Cu$K\alpha$ X-rays) $= 99{\cdot}1$ cm.$^{-1}$, $F(000) = 464$.

Absent reflexions. $h0l$ with $h + l$ odd, and $0k0$ with k odd, indicating the space group $P2_1/n$ (No. 14). This carries no implications of molecular symmetry; all atoms may be in general four-fold positions, $\pm(x, y, z;\ \tfrac{1}{2} + x, \tfrac{1}{2} - y, \tfrac{1}{2} + z)$.

Eleven reflexions observed with the rubidium salt violate the space-group conditions. Nearly all of them come into the " very-very weak " category, the exceptions being 050 and 0,13,0 which are " weak " and " very weak," respectively. Ten of these reflexions were observed in photographs taken about the *a*-axis; and that they were due to the Renninger effect was suggested by the fact that they did not appear in photographs taken about another axis. This explanation was easily validated for the two strongest of these spots; 050 can be attributed to *two* double reflexions involving two pairs of strong—or rather strong—spots, *viz.* $0\bar{4}\bar{1}/091$ and $0\bar{6}\bar{2}/0,11,2$, whilst 0,13,0 can be attributed to $04\bar{1}/091$. This interpretation is discussed in a Note to be published elsewhere.[5]

The success of the analysis was evidence that no serious error of space group was occasioned by neglect of these adventitious reflexions.

Course of the Analysis and Refinement.—Structural study was confined to the rubidium salt. Intensities were estimated visually from multiple-film exposures. Some details of the reflexions measured are included in Table 1. The crystal habit facilitated collection of values about the *a*-axis. A crystal of nearly square cross-section could be used, and absorption corrections applied as for a cylinder with $\mu R = 0{\cdot}64$. The intensity measurements in the nets $0kl$, $1kl$, and $2kl$ are therefore regarded as more accurate than those for the few additional reflexions, derived from *c*-axial photographs, to which no absorption correction was readily applicable. Observed structure amplitudes were placed on an approximately absolute scale by comparison, with calculated structure-factors during the earlier stages of the analysis; subsequently a single scale-factor was one of the parameters refined in the least-squares (L.–S.) procedure.

TABLE 1

Summary of terms used in the analysis

Reciprocal lattice net	No. of obsvd. reflexions	Percentage of those accessible	R (%)
$0kl$	216	88·2	11·17
$1kl$	428	91·1	9·15
$2kl$	362	85·6	7·74
$3,4,5kl$	33	5·7	13·53
All	1039	60·7	9·26

In its *x*-axial projection, the structure was easily solved by application of the minimum function to the sharpened Patterson projection. It was refined to $R = 13{\cdot}8\%$ by repeated electron density and difference syntheses. Approximate *x*-co-ordinates were then assessed by trial, and refined by generalised projections based on $1kl$ and $2kl$ data. Finally, all the data were used in nine cycles of L.–S. refinement, the weighting system being as described in Part VI.[6] These and all other computations were performed on the Glasgow University DEUCE the main programs were those of Rollett,[7] and the subsidiary programs were mainly due to Sime.[7]

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[5] J. C. Speakman, *Acta Cryst.*, 1965, **18**, in the press (see also R. W. James, " The Optical Principles of the Diffraction of X-Rays " (p. 25), Bell, London, 1950).

[6] J. C. Speakman and H. H. Mills, *J.*, 1961, 1164.

[7] " Computing Methods and the Phase Problem in X-Ray Crystal Analysis," Pergamon Press, Oxford, 1961.

Atomic scattering functions were taken from *International Tables*, Volume III. The function for Rb was corrected for anomalous dispersion, though the correction was small.

The hydrogen atoms were omitted from consideration until a late stage of the refinement; and then, in order to achieve an objective assessment of their positions, a three-dimensional difference synthesis was computed. Peaks of height 0·95—1·32 electrons per Å^3 indicated the positions of the four methylenic hydrogen atoms. As expected, the three hydroxylic hydrogens were less clearly defined; but regions of significant density (0·61, 1·48, and 0·70 maxima) did appear in reasonable positions. Co-ordinates were allocated so as to place these atoms 1·0 (or 1·1) Å from their respective oxygen (or carbon) atoms, and in appropriate directions, and were included in subsequent structure-factor calculations, with an isotropic Debye factor (B) of 2·29 Å^2, though these parameters were not refined. The only other significant feature of the difference synthesis was a negative region at the site of the rubidium ion. We cannot explain this, unless it be due to some untoward interaction between the vibrational parameter and the over-all scale factor. Refinement was stopped when the shifts became less than one-fifth of the standard deviations.

The final co-ordinates are listed in Table 2; the co-ordinates (in Å) with respect to orthogonal axes are accompanied in parentheses by standard deviations calculated from the L.-S. residuals. The numbering of atoms is shown in Figure 1, hydrogen atoms being designated by reference to the atom to which they are bonded. Co-ordinates are given as they were used in computation; in many cases more figures appear than are significant. Token positions are shown in Figure 1 for the hydroxylic hydrogens, to indicate to which oxygen atoms they are covalently linked. Vibrational parameters are given in Table 3 in the form of tensor-components representing the ellipsoid of mean-square amplitude. The set of atoms, including two glycollate residues, whose parameters are listed in these Tables constitute the standard chemical

TABLE 2

Rubidium hydrogen bisglycollate: fractional ($x, y, z, \times 10^5$) and orthogonal ($X', Y Z', \times 10^4$, in Å) co-ordinates. (The quantities in parentheses are the estimated standard deviations; Y and Z' are, respectively, parallel to y and z, whilst X' is perpendicular to both. For the numbering of atoms see Figure 1.)

Atom	x	x	z	X'	Y	Z'
Rb	77,710	38,262	52,374	31,148(10)	68,527(10)	50,482(10)
O(1)	33,101	48,240	64,689	13,268(100)	86,398(80)	66,087(70)
O(2)	59,049	53,550	82,392	23,669(110)	95,908(90)	83,169(80)
O(3)	8162	37,120	78,801	3,272(100)	66,482(70)	82,414(100)
O(4)	−3249	25,416	32,791	−1303(90)	45,520(70)	34,690(70)
O(5)	21,459	36,313	31,510	8602(110)	65,037(70)	31,874(80)
O(6)	−19,675	24,133	7192	−7887(120)	43,223(80)	8735(70)
C(1)	40,759	48,293	76,264	16,338(140)	86,493(100)	77,809(90)
C(2)	31,371	42,504	84,986	12,575(160)	76,125(110)	87,542(100)
C(3)	6182	30,649	26,472	2478(130)	54,893(90)	27,482(100)
C(4)	−2435	30,737	11,923	−976(130)	55,050(100)	12,688(100)
HC(2)	52,709	39,630	90,710	21,129	70,977	92,307
HC(2)	20,229	44,950	92,360	8109	80,505	95,965
HC(4)	23,549	30,039	10,100	9438	53,800	9231
HC(4)	−19,190	35,539	9800	−7693	63,650	11,146
HO(2)	66,970	57,190	76,340	26,846	102,427	76,345
HO(3)	20,189	32,320	78,720	8093	57,885	81,618
HO(6)	−33,191	24,281	−1400	−13,305	43,487	492

TABLE 3

Rubidium hydrogen bisglycollate: vibrational parameters (Å, $\times 10^4$)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{31}	Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{31}
Rb	315	331	292	11	−26	1	O(6)	767	348	235	−116	−50	−118
O(1)	392	418	254	74	2	−128	C(1)	401	285	209	118	7	36
O(2)	738	414	329	−178	12	−42	C(2)	758	321	295	−147	−13	−43
O(3)	247	304	581	−44	6	−59	C(3)	247	227	309	40	−19	37
O(4)	231	389	335	−44	36	−59	C(4)	247	305	257	−59	−44	36
O(5)	398	322	341	−61	−24	−84							

TABLE 4

Rubidium hydrogen bisglycollate: calculated structure factors and observed structure amplitudes

h	*k*	*l*	$\lvert F_o \rvert$	F_c
0	0	2	17.8	10.3
		4	53.4	59.7
		6	67.2	69.3
		8	11.7	11.8
0	1	1	55.3	82.4
		2	17.0	-14.8
		3	79.0	95.7
		4	90.3	-114.2
		5	40.2	38.1
		6	20.2	-18.7
		7	37.0	36.8
		8	32.5	-33.0
		10	29.4	-29.8
		12	19.0	-18.9
		13	7.0	-7.7
0	2	1	11.3	-9.7
		2	20.2	-20.6
		3	46.6	-49.0
		4	4.2	4.2
		5	61.6	-69.0
		6	31.1	26.4
		7	53.4	-54.8
		8	14.8	13.8
		9	56.9	-55.4
		10	9.2	-8.5
		11	35.7	-33.7
		13	14.6	-18.6
0	3	1	54.5	-66.7
		2	6.6	-4.0
		3	44.8	-55.2
		4	18.1	-14.3
		5	60.9	-66.0
		6	47.0	-43.6
		7	4.0	-4.5
		8	54.3	-57.2
		10	33.6	-32.2
		11	1.8	-4.7
		12	26.8	-28.7
0	4	0	57.8	-64.1
		1	41.6	-50.3
		2	79.5	-97.7
		3	19.1	-13.6
		4	89.4	-99.9
		5	3.4	-2.5
		6	44.8	-45.9
		7	5.0	4.4
		8	21.1	-19.8
		9	26.8	-24.0
		10	21.3	-17.3
		11	2.4	-5.3
		12	16.0	16.4
0	5	1	72.9	-87.4
		2	15.7	-16.6
		3	80.8	-89.2
		4	42.1	38.8
		5	49.8	-50.1
		6	11.8	9.3
		7	28.2	-25.6
		8	37.0	33.7
		9	14.3	-14.3
		10	31.7	29.3
		11	2.9	-4.4
		12	13.6	14.2
		13	14.4	15.5
0	6	0	24.6	-26.8
		1	41.7	35.6
		2	55.9	-60.7
		3	26.4	22.5
		4	12.1	-9.3
		5	53.9	55.6
		6	19.9	-16.9
		7	50.0	47.7
		8	10.4	9.6
		9	42.6	42.5
		11	38.1	38.3
		12	9.8	-3.7
0	7	1	56.9	60.2
		2	25.8	-21.4
		3	10.9	9.1
		4	86.6	91.5
		5	20.5	18.3
		6	54.5	55.8
		7	38.4	35.3
		8	28.2	25.9
		9	7.5	-6.6
		10	39.9	38.6
		12	14.2	15.5
0	8	0	104.2	136.5
		1	8.8	5.8
		2	54.9	62.8
		3	27.8	23.3
		4	62.1	63.2
		5	12.0	9.3
		6	49.5	44.2
		7	37.4	32.6
		8	6.1	-3.8
		11	13.3	11.5
0	9	1	83.3	97.9
		2	14.6	-12.5
		3	55.4	51.9
		4	40.5	-34.9
		5	43.0	41.1
		6	10.2	-8.8
		7	38.1	33.0
		8	25.5	-23.9
		9	4.1	-0.3
		10	7.1	-7.8
0	10	0	17.5	16.0
		1	24.1	-21.7
		2	32.9	33.4
		3	28.6	-25.3
		4	35.7	31.7
		5	61.8	-58.5
		6	4.2	3.0
		7	22.6	-19.5
		8	35.5	33.8
		9	40.6	-39.7
		11	25.8	-24.1
		12	7.2	-6.7
0	11	1	28.1	-27.5
		2	42.4	-43.5
		3	17.3	-13.7
		4	14.0	-12.3
		6	24.2	-21.4
		7	20.4	-5.5
		8	54.2	-57.3
		10	29.2	-25.4
0	12	0	55.3	-60.7
		1	8.8	-6.9
		2	36.8	-36.0
		3	6.4	-5.4
		4	54.2	-49.0
		5	15.2	-11.4
		6	28.0	-26.0
		7	27.9	-26.2
		8	10.5	-10.5
		9	21.6	-20.0
		10	10.2	-8.3
		11	11.0	-11.3
0	13	1	45.0	-46.5
		2	16.1	-13.5
		3	71.3	-61.3
		4	13.3	10.9
		5	37.0	-37.3
		7	23.8	-22.6
		8	2.4	5.5
		9	14.1	-10.5
		10	8.6	7.9
		11	6.6	7.6
0	14	0	41.2	-43.4
		1	12.7	-11.4
		2	38.2	-36.1
		3	13.9	13.0
		4	25.4	-23.7
		5	26.7	24.3
		6	19.2	-15.1
		7	36.3	34.4
		8	1.7	-4.7
		9	15.3	15.1
		10	1.6	-2.6
0	15	1	9.4	-3.2
		2	13.2	14.5
		3	3.2	1.2
		4	34.1	33.8
		5	12.2	10.8
		6	31.0	29.3
		8	27.2	24.4
		9	4.5	-3.3
		10	24.2	23.2
0	16	0	49.2	54.4
		2	25.0	23.3
		3	15.7	17.2
		4	27.4	25.0
		5	27.0	24.6
		6	13.4	10.5
		7	19.5	16.7
		8	4.6	5.9
		9	8.5	7.9
0	17	1	42.7	45.8
		2	4.1	5.2
		3	35.6	34.2
		4	21.6	-20.1
		5	23.1	21.8
		6	14.1	11.8
		7	16.5	13.7
0	18	1	19.5	-17.9
		2	36.0	39.4
		3	11.4	-9.6
		4	26.6	23.8
		5	17.2	-14.9
		6	13.8	11.3
		7	16.2	-14.9
		8	8.2	8.5
0	19	2	10.2	-8.1
		4	13.4	-12.4
		5	6.3	4.9
		6	20.1	-20.4
		7	4.7	4.7
0	20	0	22.4	-19.2
		1	17.7	-16.5
		2	12.4	-11.6
		4	13.2	-12.5
		5	8.4	-7.7
		6	9.7	-9.9
0	21	1	25.4	-22.4
		2	3.5	-4.2
		3	26.0	-25.4
		5	10.0	-10.1
0	22	0	23.4	-23.3
		1	5.8	6.0
		2	20.0	-19.0
		4	11.7	-13.7
0	23	1	8.0	-8.5
1	0	1	8.6	-8.7
		3	7.4	-5.8
		5	91.8	-91.2
		7	75.4	-71.3
		9	30.5	-27.6
		11	44.3	-49.0
		-1	26.6	-31.8
		-3	25.0	21.3
		-5	16.8	13.8
		-7	60.2	61.5
		-9	47.3	47.6
		-11	38.6	40.0
		-13	23.2	24.6
1	1	0	8.9	8.7
		1	59.9	-93.6
		2	42.7	-45.5
		3	61.0	-66.4
		4	55.8	-51.8
		5	35.8	-30.9
		6	45.4	-48.5
		7	24.9	-24.3
		8	33.4	-31.0
		9	7.5	-8.6
		10	40.4	-40.1
		11	14.6	13.2
		12	15.4	-15.1
		-1	18.2	-15.9
		-2	1.5	2.2
		-3	49.3	-50.1
		-4	14.2	12.9
		-5	67.2	-65.3
		-6	58.9	60.1
		-7	41.8	-41.4
		-8	28.3	28.7
		-9	13.6	-12.5
		-10	24.2	25.4
		-12	29.0	27.8
		-13	6.7	6.7
1	2	0	32.6	-44.6
		2	86.9	-144.5
		3	28.4	-26.9
		4	44.4	-41.7
		5	12.0	-11.6
		6	44.1	-41.7
		7	5.8	-3.8
		8	16.4	-14.7
		9	7.2	6.6
		10	5.8	4.9
		12	13.3	13.4
		-1	24.4	-19.7
		-2	93.0	-144.2
		-3	13.8	13.9
		-4	105.3	-112.0
		-5	31.8	28.7
		-6	23.7	-22.9
		-7	18.2	18.1
		-8	43.5	-40.8
		-9	16.9	-16.5
		-10	14.1	-13.7
		-11	4.0	5.2
		-13	6.6	4.9
1	3	0	67.3	101.9
		1	54.7	-61.4
		2	22.3	16.6
		3	63.2	-73.3
		4	6.7	5.1

TABLE 4 (*Continued*)

h	k	l	$\|F_o\|$	F_c
		5	38.2	-35.0
		6	46.6	42.5
		8	45.0	45.0
		9	8.6	-7.4
		10	19.3	17.7
		11	7.3	7.6
		12	16.0	17.5
		-1	95.8	-162.3
		-2	90.2	-102.5
		-3	72.2	-72.4
		-4	19.3	15.2
		-5	51.4	-44.9
		-6	36.6	-33.5
		-7	33.7	-33.7
		-8	30.6	-28.1
		-9	8.8	-8.3
		-10	25.5	-25.5
		-12	18.3	-18.5
		-13	5.1	-4.1
1	4	0	3.2	-1.0
		1	51.4	58.7
		2	38.2	-38.0
		3	91.0	95.3
		4	28.2	-25.3
		5	35.0	29.8
		6	16.6	14.8
		7	70.0	68.6
		8	4.4	-3.5
		9	39.5	41.3
		11	21.1	21.5
		12	5.4	-3.3
		-1	32.5	-34.2
		-2	12.2	-9.1
		-3	21.3	-15.8
		-4	39.1	-33.0
		-5	71.9	-71.8
		-6	11.4	-10.7
		-7	44.0	-44.3
		-9	41.4	-43.0
		-10	11.0	-10.3
		-11	29.4	-28.9
		-12	6.9	6.3
		-13	20.1	-23.9
1	5	0	25.4	22.5
		1	32.3	29.3
		2	57.9	60.7
		3	61.8	67.6
		4	42.2	41.0
		5	6.2	3.7
		6	60.0	51.5
		8	48.1	45.1
		10	26.8	25.8
		11	5.8	-5.0
		12	21.7	22.2
		-1	80.2	80.8
		-2	59.3	-57.2
		-3	67.3	67.2
		-4	3.9	-5.3
		-5	19.3	16.3
		-6	33.0	-29.7
		-7	16.9	14.3
		-8	53.0	-56.5
		-9	13.7	12.3
		-10	27.0	-27.8
		-11	8.6	-6.9
		-12	20.0	-20.5
1	6	0	95.3	107.0
		1	14.7	13.4
		2	80.0	82.1
		3	27.0	-22.4
		4	46.6	46.6
		5	13.9	13.9
		6	32.4	26.2
		7	23.4	20.6
		8	8.8	7.8
		9	16.5	15.8
		10	5.0	-4.5
		11	15.4	15.2
		12	4.9	-4.4
		-1	31.4	28.8
		-2	91.0	97.9
		-3	19.9	-16.5
		-4	63.2	61.1
		-5	8.3	-7.7
		-6	43.0	43.0
		-7	11.8	11.0
		-8	40.6	40.1
		-9	26.6	-24.8
		-10	8.8	-6.9
		-11	11.0	-10.4
1	7	0	3.8	-2.0
		1	61.8	66.6
		2	25.4	-21.2
		3	64.6	63.0
		4	22.2	-22.3
		5	25.8	24.5
		6	49.4	-44.4
		7	53.7	52.3
		9	6.0	-4.1
		10	16.5	-15.7
		11	15.2	-15.7
		12	13.4	-14.5
		-1	69.4	73.6
		-2	4.3	5.0
		-3	55.4	54.4
		-4	11.3	8.7
		-5	75.8	72.3
		-6	37.9	31.8
		-7	24.3	22.6
		-8	19.4	19.4
		-9	30.8	30.6
		-10	8.6	7.2
		-11	1.7	3.2
		-12	17.0	16.9
1	8	0	57.6	54.2
		1	16.9	-16.1
		2	9.3	-7.0
		3	37.0	-34.4
		4	29.9	29.8
		5	33.7	-29.3
		6	30.8	27.3
		7	59.3	-60.1
		8	6.2	-3.7
		9	35.7	-34.5
		10	3.3	1.5
		11	35.8	-35.1
		-1	18.6	-16.7
		-2	29.0	27.0
		-3	24.6	20.6
		-4	18.4	13.1
		-5	26.8	25.6
		-6	28.2	25.6
		-7	38.6	37.2
		-8	16.7	15.9
		-9	34.4	35.9
		-11	28.0	28.5
		-12	4.3	4.3
1	9	0	18.0	-16.1
		1	4.1	2.7
		2	15.5	-13.0
		3	35.0	-34.5
		4	41.4	-38.7
		5	17.3	-16.0
		6	68.3	-58.9
		7	9.8	-7.6
		8	31.7	-30.5
		9	6.5	-7.3
		10	32.1	-34.8
		11	11.1	10.5
		-1	18.6	-17.4
		-2	5.4	5.2
		-3	29.1	-28.2
		-4	23.3	20.8
		-5	29.4	-26.9
		-6	37.4	32.8
		-7	4.2	1.8
		-8	37.8	37.4
		-9	15.4	-13.7
		-10	27.8	30.2
		-11	4.3	-3.8
		-12	23.2	25.5
1	10	0	39.4	-36.0
		1	5.9	4.3
		2	83.1	-92.3
		3	44.6	-44.4
		4	27.7	-26.9
		5	33.8	-30.7
		6	19.3	-20.1
		7	2.1	-3.1
		8	18.5	-16.9
		9	21.0	-20.3
		10	9.6	8.4
		11	3.6	-3.8
		-1	8.9	5.4
		-2	51.7	-50.9
		-3	3.7	-2.9
		-4	65.2	-66.4
		-5	32.5	28.0
		-6	35.3	-33.2
		-7	22.5	22.9
		-8	21.1	-19.8
		-9	6.9	6.2
		-10	11.0	-8.7
		-11	16.6	15.3
		-12	4.9	5.1
1	11	0	4.9	3.8
		1	58.2	-61.0
		2	5.0	2.9
		3	51.0	-57.2
		4	5.2	4.3
		5	44.2	-39.8
		6	21.2	18.9
		7	14.6	14.2
		8	8.6	8.7
		9	13.0	-12.4
		10	7.8	6.5
		-1	68.4	-69.4
		-2	12.8	-11.8
		-3	70.7	-71.2
		-4	7.4	6.2
		-5	40.0	-36.3
		-6	12.6	-10.5
		-7	32.6	-30.7
		-8	12.3	-12.2
		-9	4.9	-5.3
		-10	8.8	-8.5
		-11	6.6	-5.2
1	12	0	34.8	-34.6
		1	31.4	28.2
		2	33.3	-29.7
		3	44.6	41.9
		4	17.0	-16.4
		5	25.4	24.7
		6	13.8	-12.8
		7	36.7	37.5
		9	25.5	22.0
		10	4.1	-3.7
		-1	19.2	-17.3
		-2	41.4	-43.4
		-3	14.9	-13.8
		-4	33.7	-28.6
		-5	23.7	-22.7
		-6	20.1	-19.7
		-7	34.9	-34.3
		-9	25.4	-26.2
		-10	12.4	-11.2
		-11	12.6	-13.9
1	13	0	23.8	22.4
		1	6.2	-2.7
		2	20.3	19.0
		3	15.4	14.6
		4	46.4	44.1
		5	7.3	-5.7
		6	26.6	28.8
		8	34.5	34.0
		10	23.4	24.0
		-1	35.6	33.8
		-2	10.6	-9.0
		-3	7.4	6.0
		-4	27.8	-25.2
		-5	3.9	-3.2
		-6	43.6	-41.3
		-7	5.0	4.2
		-8	15.8	-13.8
		-10	26.6	-27.4
		-11	7.4	-7.8
1	14	0	46.6	46.0
		1	13.0	11.8
		2	38.2	35.4
		3	6.3	5.1
		4	10.8	10.6
		5	21.2	23.7
		6	24.9	23.9
		7	18.2	18.4
		9	20.8	18.2
		-1	11.8	5.6
		-2	50.1	49.1
		-3	14.3	-13.4
		-4	34.6	33.4
		-6	11.4	9.4
		-7	16.1	-14.5
		-8	25.6	21.1
		-9	24.4	-23.6
1	15	0	16.7	-18.2
		1	44.9	42.8
		2	9.0	-8.5
		3	35.9	34.6
		5	23.2	25.8
		6	6.0	-4.5
		7	16.4	16.6
		9	1.9	-2.6
		-1	46.3	48.2
		-2	24.0	25.7
		-3	32.9	33.8
		-4	9.6	-8.2
		-5	39.0	37.4
		-6	12.1	10.7
		-7	25.0	24.8
		-8	3.5	3.0

TABLE 4 (*Continued*)

h	k	l	$\|F_o\|$	F_c
		- 9	11.8	11.0
1	16	0	45.6	43.5
		2	17.6	18.4
		3	14.5	- 15.1
		4	18.2	18.3
		5	22.0	- 23.2
		6	19.0	18.2
		7	18.9	- 18.4
		8	7.9	8.1
		- 2	13.1	13.0
		- 3	2.8	3.0
		- 4	23.4	23.2
		- 5	15.1	14.0
		- 6	28.6	27.0
		- 7	15.6	14.0
		- 8	13.0	12.2
		- 9	20.0	20.9
1	17	0	13.1	- 11.0
		1	9.3	- 9.6
		2	9.7	- 10.5
		4	18.5	- 20.1
		5	5.8	5.0
		6	28.9	- 28.2
		8	20.0	- 23.4
		- 1	22.5	20.6
		- 2	6.8	7.7
		- 4	12.5	12.5
		- 6	16.2	14.0
		- 8	21.2	23.8
1	18	0	11.6	- 10.9
		1	6.2	- 7.4
		2	23.3	- 25.9
		3	21.1	- 23.1
		4	14.4	- 14.5
		5	10.9	- 11.1
		6	11.6	- 10.1
		7	15.5	- 15.0
		- 2	22.4	- 23.3
		- 3	6.9	6.3
		- 4	22.2	- 19.9
		- 5	19.6	18.8
		- 6	15.1	- 12.5
		- 7	10.2	9.5
		- 8	5.9	- 5.4
1	19	1	28.0	- 28.1
		2	2.5	- 4.8
		3	23.2	- 23.3
		4	6.2	- 6.5
		5	16.4	- 16.3
		- 1	33.3	- 36.5
		- 2	6.2	- 5.6
		- 3	30.7	- 28.4
		- 4	7.8	7.4
		- 5	24.5	- 23.2
		- 7	7.7	- 8.8
1	20	0	24.8	- 27.9
		2	15.6	- 16.5
		3	15.4	14.3
		4	20.1	- 20.6
		5	8.8	8.4
		- 2	23.3	- 22.9
		- 3	5.5	- 5.3
		- 4	16.6	- 15.6
		- 5	15.2	- 14.6
		- 6	17.7	- 20.1
1	21	0	3.1	- 3.3
		1	11.6	- 12.4
		2	15.6	15.7
		3	3.6	- 1.5
		4	16.2	19.0
		- 1	9.3	6.5
		- 3	11.9	- 11.0
		- 4	11.2	- 12.0
1	22	0	10.6	11.5
		1	1.0	1.4
		2	11.6	12.0
		- 1	6.2	6.6
		- 2	10.1	10.5
		- 3	4.7	- 5.9
2	0	0	72.2	- 73.3
		2	52.1	- 54.0
		4	49.1	- 44.8
		6	15.8	- 13.8
		8	4.3	4.3
		10	8.2	8.6
		- 2	103.9	-139.7
		- 4	47.8	- 49.8
		- 6	63.8	- 63.7
		- 8	22.3	- 20.8
		-10	11.7	- 11.7
		-12	7.0	- 4.6
2	1	0	24.2	21.8
		1	51.5	- 60.1
		2	54.4	54.6
		3	27.0	- 19.3
		4	30.2	24.1
		5	34.7	- 34.8
		6	47.7	49.3
		7	12.6	- 11.8
		8	27.1	25.4
		9	13.4	15.0
		10	15.0	13.6
		- 1	65.4	- 83.0
		- 2	17.0	11.4
		- 3	47.7	- 42.5
		- 4	35.0	- 32.3
		- 5	54.4	- 54.1
		- 6	29.9	- 30.0
		- 7	24.2	- 24.1
		- 8	22.6	- 25.3
		- 9	26.4	- 28.0
		-10	20.7	- 20.6
		-12	10.4	- 11.2
2	2	0	22.1	- 26.9
		1	33.8	31.9
		2	18.2	18.3
		3	69.6	70.4
		4	11.6	- 9.7
		5	60.4	53.8
		6	22.8	- 22.7
		7	38.5	37.8
		8	4.3	5.3
		9	45.4	49.8
		11	22.6	21.9
		- 1	16.1	17.6
		- 2	5.2	- 5.8
		- 3	12.4	- 13.2
		- 4	26.8	21.8
		- 5	25.8	- 23.2
		- 6	13.5	- 12.3
		- 7	31.8	- 32.9
		- 8	24.3	- 23.9
		- 9	35.8	- 41.4
		-10	9.4	7.4
		-11	27.3	- 28.3
		-13	16.6	- 21.2
2	3	0	19.9	19.0
		1	54.5	60.7
		2	13.7	12.3
		3	25.6	26.4
		4	55.0	52.5
		5	18.4	16.2
		6	47.0	40.6
		7	4.4	- 4.9
		8	37.1	38.5
		9	3.2	6.6
		10	28.1	31.6
		- 1	42.0	44.7
		- 2	22.0	21.1
		- 3	43.8	43.0
		- 4	28.9	- 25.8
		- 5	39.4	37.2
		- 6	12.8	- 13.9
		- 7	31.0	30.6
		- 8	16.1	- 14.8
		- 9	6.1	7.2
		-10	37.7	- 41.4
		-11	10.2	7.9
		-12	18.6	- 19.3
2	4	0	75.3	88.9
		1	6.8	3.8
		2	52.2	49.8
		3	22.0	15.4
		4	53.6	51.4
		5	6.8	- 4.6
		6	21.0	20.7
		7	24.2	21.4
		8	6.0	6.6
		9	11.0	9.8
		10	11.6	- 10.2
		- 1	4.2	2.3
		- 2	44.7	54.2
		- 3	4.6	2.7
		- 4	58.1	56.0
		- 6	75.9	77.1
		- 7	17.7	- 17.9
		- 8	17.4	18.2
		-10	18.2	18.6
		-11	6.6	- 4.9
		-12	9.2	7.1
2	5	0	33.8	35.8
		1	61.6	66.8
		2	28.5	- 28.1
		3	37.2	35.8
		4	37.7	- 34.4
		5	47.8	45.8
		6	18.9	- 16.3
		7	11.0	- 9.4
		8	27.6	- 26.4
		10	26.4	- 26.9
		11	9.4	- 9.5
		- 1	40.1	41.8
		- 2	40.5	- 45.9
		- 3	69.6	78.4
		- 4	14.6	14.5
		- 5	49.9	49.8
		- 6	20.6	20.7
		- 7	35.3	33.4
		- 8	10.2	9.8
		- 9	14.0	13.1
		-10	18.6	19.4
		-11	13.5	12.9
		-12	14.0	15.0
2	6	0	23.0	19.5
		1	7.6	1.8
		2	37.9	41.0
		3	41.0	- 40.8
		4	3.8	- 2.5
		5	67.0	- 67.7
		6	18.9	- 18.3
		7	38.2	- 32.5
		8	16.8	16.3
		9	37.6	- 41.0
		11	17.8	- 20.6
		- 1	43.6	- 50.1
		- 2	13.4	11.5
		- 3	18.4	18.8
		- 4	45.4	40.6
		- 5	22.1	20.2
		- 6	5.0	2.2
		- 7	35.1	35.5
		- 8	8.2	8.0
		- 9	25.0	26.4
		-11	25.9	28.2
2	7	0	7.0	- 8.0
		1	34.0	- 33.4
		2	67.9	- 66.9
		3	3.8	- 0.5
		4	48.6	- 44.6
		5	31.1	- 31.6
		6	36.6	- 36.9
		8	34.8	- 32.7
		9	7.4	8.4
		10	18.9	- 19.2
		- 1	36.7	- 36.8
		- 2	6.7	3.1
		- 3	13.3	- 13.2
		- 4	13.8	10.4
		- 5	31.8	- 31.8
		- 6	35.5	32.6
		- 7	17.0	- 13.4
		- 8	28.6	30.7
		- 9	9.8	- 8.4
		-10	19.8	21.3
		-12	18.5	22.7
2	8	0	68.7	- 67.2
		1	23.6	- 21.4
		2	37.9	- 43.7
		3	12.2	- 13.4
		4	34.0	- 29.3
		5	25.9	- 24.4
		6	5.0	- 5.3
		7	9.9	- 8.1
		9	8.6	- 8.8
		- 2	72.5	- 78.2
		- 4	44.9	- 46.5
		- 5	5.0	5.0
		- 6	37.6	- 33.9
		- 7	17.3	14.8
		- 8	26.7	- 28.6
		- 9	11.7	11.1
		-10	9.8	- 9.1
2	9	0	22.9	20.2
		1	51.9	- 51.6
		2	25.3	25.5
		3	32.8	- 32.1
		4	19.4	17.4
		5	22.2	- 22.6
		6	8.2	5.9
		7	7.0	- 5.9
		8	17.8	16.0
		- 1	66.0	- 68.5
		- 2	18.6	- 22.2
		- 3	51.0	- 58.2
		- 4	9.7	- 7.8
		- 5	47.4	- 45.4
		- 6	9.4	- 5.5
		- 7	28.8	- 28.0
		- 8	15.0	- 12.8

TABLE 4 (*Continued*)

h	k	l	$\lvert F_o \rvert$	F_c
		-9	20.2	-20.2
		-10	16.6	-16.8
2	10	0	16.0	-16.3
		1	22.6	22.5
		2	23.9	-20.9
		3	37.0	36.7
		4	14.1	-14.1
		5	33.0	31.8
		6	24.2	-24.2
		7	36.4	37.3
		8	2.2	4.1
		9	24.7	26.3
		10	6.6	5.8
		-1	28.9	27.6
		-2	22.6	-25.3
		-3	18.7	-19.5
		-4	20.1	-20.9
		-5	22.1	-19.2
		-6	26.3	-26.4
		-7	22.8	-23.9
		-8	11.5	-9.0
		-9	21.1	-23.4
		-10	10.7	-9.7
		-11	20.2	-21.0
2	11	0	19.2	18.2
		1	28.3	29.0
		2	25.4	25.4
		3	5.5	-7.4
		4	31.0	29.8
		5	13.7	12.7
		6	39.3	39.3
		7	6.1	-4.9
		8	35.9	35.3
		9	3.3	-0.7
		-2	6.1	5.4
		-3	17.1	16.4
		-4	9.7	-9.7
		-5	21.8	21.2
		-6	18.5	-19.9
		-8	20.7	-19.8
		-10	27.2	-28.9
		-11	4.9	4.3
2	12	0	33.4	36.3
		1	18.4	20.4
		2	21.3	22.3
		3	15.9	14.7
		4	36.7	36.6
		5	13.3	13.3
		6	10.8	10.9
		7	27.9	25.7
		9	11.0	11.5
		-2	43.6	50.7
		-4	23.8	23.0
		-5	8.3	-7.4
		-6	37.6	38.6
		-8	15.6	14.3
		-9	16.2	-17.5
		-10	9.0	8.2
2	13	0	20.6	21.4
		1	50.8	52.3
		3	27.9	27.8
		4	11.1	-12.2
		5	9.8	11.5
2	13	7	16.5	15.7
		9	4.2	-4.7
		-1	39.1	43.6
		-2	25.6	-25.6
		-3	32.2	31.1
		-4	10.9	11.0
		-5	38.7	40.5
		-6	5.4	5.2
		-7	26.8	27.9
		-9	15.0	15.1
		-10	5.7	6.5
2	14	0	14.2	14.3
		1	11.5	-9.5
		2	25.3	26.0
		3	21.6	-22.7
		4	19.0	17.5
		5	32.4	-32.4
		7	13.8	-11.8
		-2	39.3	37.8
		-4	28.9	30.1
		-5	14.4	11.8
		-6	9.4	7.9
		-7	12.3	12.5
		-8	17.9	15.8
		-9	16.2	18.2
		-10	4.6	5.6
2	15	0	6.2	-4.6
		2	34.2	-33.7
		4	23.0	-22.7
		5	10.2	-9.2
		6	32.3	-33.8
		7	4.8	4.6
		8	12.8	-18.2
		-1	7.9	-6.8
		-2	7.0	-7.3
		-4	11.1	9.8
		-6	19.8	19.4
		-7	4.6	-4.1
		-8	23.7	24.5
2	16	0	18.4	-17.9
		1	18.0	-17.9
		2	26.2	-25.8
		3	12.7	-13.9
		4	13.4	-12.2
		5	20.0	-20.5
		7	17.0	-17.1
		-1	4.9	-4.5
		-2	33.1	-32.9
		-4	24.2	-22.6
		-5	11.4	8.4
		-6	21.1	-19.8
		-7	16.9	17.7
2	17	1	29.0	-30.6
		2	5.0	4.1
		3	20.8	-22.5
		4	8.2	-8.5
		5	10.2	-9.8
		6	6.5	6.0
		-1	28.8	-29.7
		-2	4.0	0.7
		-3	37.5	-38.4
		-5	26.5	-24.2
		-6	6.2	-6.2
		-7	15.6	-15.6
		-8	3.4	3.5
2	18	0	23.0	-23.7
		1	12.0	12.0
		2	17.9	-18.1
		3	14.2	15.4
		4	10.3	-10.0
		5	12.7	12.0
		-1	6.9	6.7
		-2	14.3	-16.2
		-3	12.1	-12.1
		-4	25.2	-22.9
		-6	11.8	-11.7
		-7	8.7	-10.4
2	19	0	12.4	11.4
		1	3.8	-1.3
		2	14.6	13.8
		3	4.6	-5.4
		4	19.1	19.1
		-4	3.4	-1.8
		-6	10.2	-12.4
2	20	0	18.4	18.5
		1	8.6	7.8
		2	6.9	6.2
		3	8.0	7.7
		-1	6.8	7.9
		-2	11.7	11.5
		-3	4.6	-4.1
		-4	5.5	6.5
2	21	0	5.5	5.8
		1	12.7	14.6
		-1	15.8	17.7
		-2	5.7	-5.6
3	1	0	13.0	9.7
3	2	0	57.9	58.6
3	3	0	29.0	-23.4
3	4	0	17.9	13.4
3	5	0	41.1	-39.9
3	6	0	41.8	-42.3
3	7	0	20.0	18.8
3	8	0	10.6	-9.6
3	9	0	18.5	16.6
3	10	0	32.2	42.0
3	11	0	8.5	-11.2
3	12	0	21.5	28.0
3	13	0	18.6	-16.9
3	14	0	20.5	-26.5
3	16	0	12.0	-13.1
3	17	0	12.6	12.9
3	18	0	12.8	14.3
4	0	0	37.8	33.8
4	1	0	29.4	-18.1
4	2	0	10.0	-10.1
4	3	0	23.3	-23.6
4	4	0	30.8	-28.3
4	5	0	11.0	14.2
4	6	0	12.6	-12.9
4	7	0	22.0	19.4
4	8	0	19.9	21.8
4	10	0	5.6	7.4
4	11	0	15.4	-21.0
4	12	0	12.9	-15.3
5	1	0	16.1	-13.7
5	2	0	14.4	-13.0
5	3	0	9.8	9.7

unit (S.C.U.). When necessary in the discussion, neighbouring symmetry-related units are designated as follows:

S.C.U.	x	y	z	III	$1 - x$	$1 - y$	$1 - z$
I	$1 + x$	y	z	IV	$1\frac{1}{2} + x$	$\frac{1}{2} - y$	$\frac{1}{2} + z$
II	$\frac{1}{2} + x$	$\frac{1}{2} - y$	$\frac{1}{2} + z$				

The mean standard deviations in the positions of the various types of atoms were taken to be as follows: Rb$^+$ 0·0010, O 0·0087, C 0·014 Å. For interatomic distances these lead to Rb$^+ \cdots$ O 0·009, C–O 0·014, C–C 0·016, and O $\cdots$ O 0·012 Å; and for angles, C–C–O 1·0°, C–O $\cdots$ O 0·9°. Structure-factors calculated with the parameters listed are compared with observed structure amplitudes in Table 4. R-values, for the observed reflexions only, are included in Table 1. In the three main reciprocal-lattice nets, structure-factors were also calculated for the accidentally absent, but accessible, reflexions. Only 21 terms, gave a value of $|F_c|$ more than three times greater than the minimum $|F_o|$ observable. The four largest terms were: 2,8,$\overline{11}$ (5·12); 2,17,$\bar{4}$ (−4·72); 2,15,$\bar{3}$ (4·48); and 1,4,10 (−4·48).

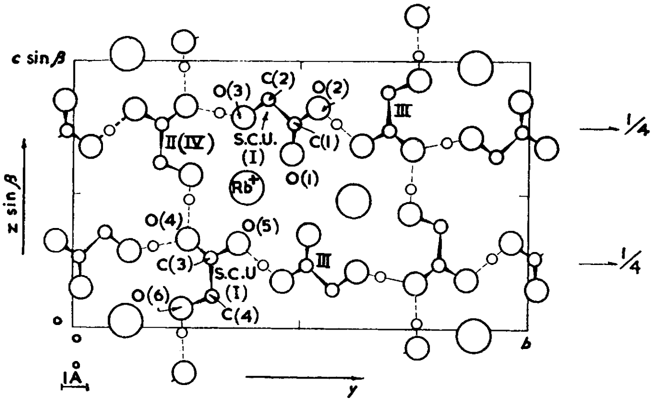


FIGURE 1. Rubidium hydrogen bisglycollate: view of the unit cell in its *a*-axial projection. (S.C.U. indicates the standard chemical unit, the co-ordinates of whose atoms appear in Table 2; other symmetry-related units are indicated by Roman numerals as is explained in the text)

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

A general impression of the structure can be obtained from Figure 1. The more important interatomic distances and angles are listed in Table 5. These dimensions have not been corrected for the effects of torsional oscillation. The glycollate residues are anchored at each end, either by electrovalencies or by hydrogen bonds, and are unlikely to be executing rigid-body librations of any great amplitude. This is supported by inspection of the anisotropic vibrational parameters in Table 3. We conclude that any corrections to the bond-lengths would not exceed 0·002 Å, and so are insignificant.

TABLE 5

Interatomic distances (Å), and bond-angles

C(1)–O(1)	1·212	C(3)–O(4)	1·241	O(2)H · · · · O(5$^{\rm III}$)	2·529
C(1)–O(2)	1·308	C(3)–O(5)	1·264	O(6$^{\rm II}$)H · · · O(4)	2·724
C(2)–O(3)	1·435	C(4)–O(6)	1·426	O(3)H · · · · O(4$^{\rm II}$)	2·733
C(1)–C(2)	1·471	C(3)–C(4)	1·519		
Rb · · · O(1)	2·971	Rb · · · O(1$^{\rm I}$)	3·009	Rb · · · O(3$^{\rm I}$)	2·879
Rb · · · O(5)	2·944	Rb · · · O(5$^{\rm I}$)	3·037	Rb · · · O(1$^{\rm III}$)	3·005
Rb · · · O(6$^{\rm II}$)	3·026	Rb · · · O(6$^{\rm IV}$)	3·068	Rb · · · O(4$^{\rm I}$)	3·256
O(1)–C(1)–O(2)	123·0°	O(4)–C(3)–O(5)	123·5°		
O(1)–C(1)–C(2)	124·8	O(4)–C(3)–C(4)	120·3		
O(2)–C(1)–C(2)	112·3	O(5)–C(3)–C(4)	116·1		
O(3)–C(2)–C(1)	113·8	O(6)–C(4)–C(3)	111·8		
C(1)–O(2)H · · · · · O(5$^{\rm III}$)	115·7°				
C(4$^{\rm II}$)–O(6$^{\rm II}$)H · · · O(4)	117·1				
C(2)–O(3)H · · · · · O(4$^{\rm II}$)	99·2				

In acid salts of class *A*, the two acidic residues of the gross formula MHY_2 are crystallographically equivalent. They cannot be distinguished as H*Y* and Y^-, and this is the structural criterion for allocating such acid salts to this class. That rubidium hydrogen bisglycollate belongs to class *B* was indicated by its spectrum, and this received some confirmation from the space-group implication that its two glycollate residues are non-equivalent. One residue should then be recognisable as the neutral molecule H*Y*, and the other as the anion Y^-; this distinction can be made. First, the dimensions of the carboxylate groups differ: the C–O distances are substantially equal, and the C–C–O angles differ only by 4°, in one, which must therefore be the anion; whilst the C–O distances differ very significantly, and the C–C–O angles by 12°, in the other, which must be the neutral molecule. Secondly, our location of the relevant hydrogen atom (see Figure 1) is sufficiently reliable to prove O(2) to be hydroxylic. We conclude that atoms C(1), C(2), O(1), O(2), and O(3) constitute HY, and that C(3), C(4), O(4), O(5), and O(6) Y^-.

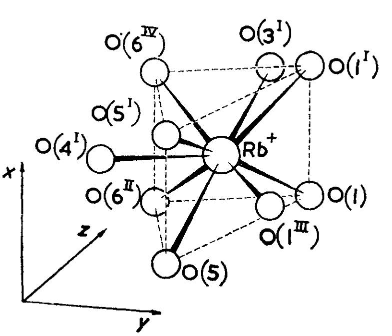
The five carbon/oxygen atoms of Y^- do not deviate significantly from the plane.

$$0{\cdot}8671X' - 0{\cdot}4624Y - 0{\cdot}1856Z' = -2{\cdot}8514 \text{ (Å)}.$$

The corresponding atoms of HY are not coplanar. This can be ascribed to twisting of the molecule through some 7° about the C–C bond; for the atoms C(1), C(2), O(1), and O(2) do not deviate significantly from the plane $0{\cdot}8295X' - 0{\cdot}5136Y - 0{\cdot}2194Z' = -4{\cdot}7885$ (Å), whilst O(3) lies 0·162 Å from the plane. The C–C distances in this residue is 0·048 Å shorter than that in the planar Y^- residue. But the difference is less than three times its standard deviation, and may thus not be significant.

There are three distinct hydrogen bonds in this structure, one short and strong, the other two long and rather weak. The strong bond links HY to Y^-, and has the favourable value of 116° for the angle C(1)–O(2) $\cdots$ O(5^{III}), and O(2) $\cdots$ O(5^{III}) = 2·529 Å. Though short, this bond is appreciably longer than the symmetrical hydrogen bonds found in some acid salts of type *A* (see the following Paper). It completes a recognisable HY_2^- complex. The weaker bonds are both between a hydroxyl and a carboxyl group, and they link the HY_2^- units into an infinite three-dimensional array. One of these bonds connects glycollate anions into chains in the [101], or [$10\bar{1}$], direction, successive units along the chain being repeated by operation of the *n*-glide; it has O(4) $\cdots$ O(6^{II}) = 2·724 Å, though the C(4^{II})–O(6^{II}) $\cdots$ O(4) angle of 117° would favour stronger bonding. The other weak bond, with O(3) $\cdots$ O(4^{II}) = 2·733 Å and the much less favourable C(2)–O(3) $\cdots$ O(4^{II}) angle of 99°, links the HY_2^- complexes into chains which spiral round the 2_1-axis parallel to [*b*].

FIGURE 2. Environment of the rubidium ion



Pairs of rubidium ions, related by centres of symmetry, are 4·05 and 4·76 Å apart. The closer pair is well cushioned by oxygen atoms. The environment of the Rb$^+$ ion is sketched in Figure 2; eight oxygen atoms are involved and they belong to six different glycollate residues. Six Rb $\cdots$ O contacts are to three pairs of oxygen atoms, each pair consisting of two oxygens related by the *a*-translation; these oxygen atoms constitute an irregular trigonal prism; with Rb $\cdots$ O distances ranging from 2·94 to 3·07 Å. Two other contacts are to oxygen atoms in directions through the larger side-faces of this prism; one of these is the shortest of all the contacts (2·88 Å), and this is to the hydroxylic oxygen atom which, as explained above, is pulled out of the plane of the other carbon or oxygen atoms of the HY molecule.

Only two non-bonded intermolecular contacts are noteworthy: these involve distances of $3{\cdot}32_5$ and 3·35 Å between O(2) and, respectively, C(3^{III}) and C(4^{III}) of a neighbouring anion.

No other glycollic acid derivatives appear to have been studied structurally. Glycine has been intensively studied,[8] but the analogy between this molecule and that of glycollic acid is vitiated by its zwitterionic nature. The diglycine hydrochloride studied by Hahn and Buerger [9] is a basic salt formally antithetical to rubidium hydrogen bisglycollate.

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