The Crystal Structure of Rubidium Seleniumtriselenocyanate Hemihydrate

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The crystal structure of rubidium selenium-triselenocyanate hemihydrate, $RbSe(SeCN)_3 \cdot \frac{1}{2}H_2O$, has been determined by X-ray methods, and refined by full matrix least squares procedures. The crystals are triclinic, space group $P\bar{1}$ (No. 2), with $a = 9.180(2)$ Å, $b = 13.227(3)$ Å, $c = 9.144(2)$ Å, $\alpha = 83.89(2)°$, $\beta = 100.02(2)°$, $\gamma = 74.97(2)°$, and four formula units per unit cell.

In the crystals, the seleniumtriselenocyanate ions are dimerized. The dimensions of the dimerized unit are nearly the same as in the corresponding potassium salt. The eight selenium atoms of the unit are approximately co-planar, and the six cyano groups are located on the same side of the plane.

Each of the two central selenium atoms has a square-planar coordination with two long Se − Se bonds *trans* to two short Se − Se bonds. The four rather short Se − Se bonds are from 2.391(2) to 2.409(2) Å, and the long ones from 3.127(2) to 3.331(2) Å. The short ones are definitely longer than the Se − Se single-bond distance 2.34 Å, and the long ones are shorter than van der Waals approaches. The total lengths of these rather unsymmetrical, approximately linear three-selenium systems (the sum of the length of the two Se − Se bonds) are 5.536 to 5.737 Å which are a little longer than in the corresponding potassium salt.

Short Se⋯Se contacts, from 3.37 Å upwards, occur between dimerized units.

The seleniumtriselenocyanate ion was first isolated in 1884 − 1886 in the form of the potassium salt,[1] as one of the oxydation products of aqueous potassium selenocyanate. Later, rubidium seleniumtriselenocyanate hemihydrate, $RbSe(SeCN)_3 \cdot \frac{1}{2}H_2O$, was isolated from the reaction between aqueous rubidium cyanide and selenium diselenocyanate.[2] Its crystal structure, reported here, has been determined

as part of a study of asymmetric linear three-selenium systems. The crystal structure of the corresponding potassium salt has been reported earlier.[3]

CRYSTAL DATA

Preparative and crystallographic data on rubidium seleniumtriselenocyanate hemihydrate have been reported earlier.[2] The space group is $P\bar{1}$ (No. 2), $a = 9.180(2)$ Å, $b = 13.227(3)$ Å, $c = 9.144(2)$ Å, $\alpha = 83.89(2)°$, $\beta = 100.02(2)°$, $\gamma = 74.97(2)°$, $z = 4$. $D_x = 3.11$ g/cm³, $D_m = 3.12$ g/cm³. The present cell parameters have been chosen to get a better comparison between this structure and that of the corresponding potassium salt.[3] They are derived, from what has been reported earlier,[2] by interchanging the a and c axes and giving the b axis opposite direction.

Collection of X-ray data was done by the same methods as for the corresponding potassium salt, using Nb-filtered $MoK\alpha$ radiation, $\lambda(\alpha_1) = 0.70926$ Å. Number of reflections recorded with measurable intensities, 2664 out of 5075 possible within $\theta = 28°$. $\mu = 200$ cm⁻¹. The cell parameters were evaluated from the settings of 21 resolved reflections. The crystals used for data collection had the following dimensions, given as distances to faces from an arbitrarily chosen point; to (001) and (00$\bar{1}$), 0.082 mm; to (010) and (0$\bar{1}$0), 0.065 mm; to (011), 0.077 mm; to (0$\bar{1}\bar{1}$), 0.068 mm; to (01$\bar{1}$), 0.079 mm; to (10$\bar{1}$) and ($\bar{1}$01), 0.086 mm.

There was no decrease in the net count of the reference reflections during the collection period.

Fifteen reflections which showed extraordinarily high, or erratic, background count were at a later stage omitted from the data.

Intensities were corrected for absorption[4] and extinction.[5]

Table 1. Atomic coordinates in fractions of triclinic cell edges. Isotropic thermal parameters ($Å^2$) in the form $\exp[-8\pi^2 U(\sin^2\theta/\lambda^2)]$. Standard deviations from the least squares refinement in parentheses.

	x	y	z	U
Se_1	$-0.02749(14)$	$0.13985(9)$	$-0.14850(13)$	
Se_2	$-0.01134(14)$	$0.12233(9)$	$0.11893(13)$	
Se_3	$-0.26734(15)$	$0.10719(11)$	$0.11809(13)$	
Se_4	$0.64421(14)$	$0.11827(9)$	$0.52315(14)$	
Se_5	$0.37849(13)$	$0.13340(8)$	$0.52197(13)$	
Se_6	$0.38571(15)$	$0.12699(10)$	$0.78550(14)$	
Se_7	$0.33122(15)$	$0.12897(10)$	$0.15988(14)$	
Se_8	$0.01767(15)$	$0.13765(10)$	$0.48366(14)$	
C_1	$-0.0234(13)$	$0.2780(9)$	$-0.1718(13)$	$0.037(3)$
C_3	$-0.3497(14)$	$0.2449(10)$	$0.1307(14)$	$0.043(3)$
C_4	$0.6240(14)$	$0.2596(9)$	$0.5293(13)$	$0.038(3)$
C_6	$0.3674(14)$	$0.2682(10)$	$0.7830(14)$	$0.040(3)$
C_7	$0.2480(12)$	$0.2687(9)$	$0.1545(12)$	$0.029(3)$
C_8	$-0.0189(13)$	$0.2801(9)$	$0.4423(13)$	$0.033(3)$
N_1	$-0.0145(12)$	$0.3634(8)$	$-0.1849(12)$	$0.051(3)$
N_3	$-0.3969(14)$	$0.3298(9)$	$0.1428(13)$	$0.064(3)$
N_4	$0.6105(13)$	$0.3473(9)$	$0.5313(12)$	$0.057(3)$
N_6	$0.3546(12)$	$0.3568(9)$	$0.7814(12)$	$0.053(3)$
N_7	$0.1933(11)$	$0.3574(8)$	$0.1542(11)$	$0.042(3)$
N_8	$-0.0391(11)$	$0.3700(8)$	$0.4196(11)$	$0.044(3)$
O	$0.6098(10)$	$0.4735(10)$	$0.8190(10)$	$0.059(3)$
Rb_1	$0.24627(14)$	$0.46638(9)$	$0.43210(14)$	
Rb_2	$0.83597(15)$	$0.46082(10)$	$0.09079(14)$	

THE STRUCTURE ANALYSES

The structure was solved by Patterson and Fourier methods and by reference to the structure of $KSe(SeCN)_3 \cdot \frac{1}{2}H_2O$.[3] The x and z coordinates of the selenium atoms in the present structure could be derived from the coordinates in the potassium salt by subtracting about 0.08 cell edge in x and z coordinates. The y coordinates are about the same in the two

structures. The R-value in the final full-matrix least squares refinement was 0.043. The Fourier difference map based on these data showed no higher peak than 0.9 e/$Å^3$.

Computational procedure and programs used are described elsewhere.[5] The final atomic coordinates and temperature parameters are listed in Tables 1 and 2.

The structure factors are available from the author on request.

Table 2. Anisotropic thermal parameters ($Å^2$) in the form $\exp[-2\pi^2(h^2a^{-2}U_{11}+\cdots2hka^{-1}b^{-1}U_{12}+\cdots)]$. All values have been multiplied by 10^4. Standard deviations from least squares refinement in parentheses.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
Se_1	$375(7)$	$323(7)$	$253(7)$	$-148(6)$	$-51(5)$	$54(5)$
Se_2	$315(7)$	$316(6)$	$267(7)$	$-147(5)$	$-43(5)$	$74(5)$
Se_3	$371(8)$	$487(8)$	$514(9)$	$-242(6)$	$-156(6)$	$148(6)$
Se_4	$245(6)$	$288(6)$	$426(8)$	$-86(5)$	$-103(5)$	$87(5)$
Se_5	$248(6)$	$237(6)$	$302(7)$	$-65(5)$	$-67(5)$	$68(5)$
Se_6	$428(8)$	$326(7)$	$301(7)$	$-137(6)$	$-67(5)$	$85(6)$
Se_7	$331(7)$	$307(6)$	$366(8)$	$-76(5)$	$-76(5)$	$48(6)$
Se_8	$342(7)$	$366(7)$	$380(8)$	$-115(6)$	$-42(6)$	$74(6)$
Rb_1	$338(7)$	$308(6)$	$319(7)$	$-80(5)$	$-63(5)$	$59(5)$
Rb_2	$408(7)$	$374(7)$	$313(7)$	$-146(6)$	$-79(5)$	$102(6)$
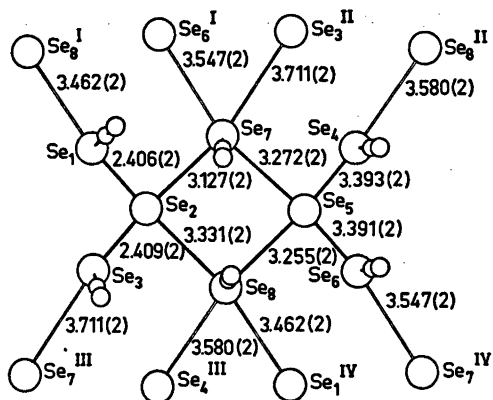
Fig. 1. The di-μ-selenocyanato-bis[diseleno-cyanatoselenate(II)] anion in $RbSe(SeCN)_3$ $\frac{1}{4}H_2O$, as seen normal to the *ac* plane.

RESULT

The di-μ-selenocyanato-bis[diselenocyanato-selenate(II)] anion. Bond lengths, close contacts, and angles in the anion, based on the atomic coordinates of Table 1, are given Fig. 1 and Tables 3 and 4. Distances from the rubidium ions and from the water molecule to neighbouring atoms are given in Table 6. The uncertainties in cell dimensions are taken into account in the given standard deviations. In the tables, superscript I denotes an atom at $(x,y,z-1)$, II at $(1+x,y,z)$, III at $(x-1,y,z)$, IV at $(x,y,1+z)$, V at $(\bar{x},\bar{y},\bar{z})$, VI at $(1-x,\bar{y},1-z)$, VII at $(\bar{x},\bar{y},1-z)$, VIII at $(1-x,1-y,1-z)$, IX at $(\bar{x},1-y,\bar{z})$, X at $(1-x,1-y,\bar{z})$, XI at $(\bar{x},1-y,1-z)$, where x, y, z are the coordinates of Table 1.

As in the crystals of the corresponding potassium salt, the selenium atoms occur in layers parallel to the *ac* plane at y approximately 0.127 and -0.127. Two layers, interrelated through symmetry centers at $y=0$, make a double layer. The cyano groups are located on the outsides of the double layer, the rubidium ions and the water oxygen have y approximately $\frac{1}{4}$ and are therefore located between double layers.

To show the resemblance of the layers in the two structures, Table 5, with a monoclinic coordinate system, has been worked out. The double layers in the two structures are almost identical, but from the difference in the direction of the *b* axes it is seen that the stacking of the double layers are different.

Some of the differences in bond lengths, close contacts, and angles will be underlined here. The short selenium-selenium bond lengths at the central selenium atoms, Se_2 and Se_5, are in mean 0.007 Å shorter in the present structure. This difference is uncertain since the standard deviation on the Se−Se bonds is 0.002 Å. The long selenium-selenium bonds at Se_2 and Se_5 are in the mean 0.07 Å longer. In the mean, the total length of the unsymmetrical three-selenium system, the sum of the lengths of the two Se−Se bonds, are 0.06 Å longer. This must mainly be explained as caused by the different packing in the two crystals.

In the structure of the potassium salt, the two Se−C bond lengths in the selenocyanate groups at Se_1 and Se_4 were, with some uncer-

Table 3. Angles (°) in the plane of the eight selenium unit. Standard deviations in parentheses.

$Se_1-Se_2-Se_3$	97.55(7)	$Se_4-Se_5-Se_6$	99.32(7)
$Se_3-Se_2-Se_8$	86.69(6)	$Se_6-Se_5-Se_8$	86.94(6)
$Se_8-Se_2-Se_7$	87.40(5)	$Se_8-Se_5-Se_7$	86.31(5)
$Se_7-Se_2-Se_1$	88.80(6)	$Se_7-Se_5-Se_4$	87.23(6)
$Se_1-Se_2-Se_8$	170.69(6)	$Se_4-Se_5-Se_8$	172.64(6)
$Se_3-Se_2-Se_7$	173.20(6)	$Se_6-Se_5-Se_7$	172.60(6)
$Se_2-Se_1\cdots Se_7^{I}$	169.71(5)	$Se_2-Se_7\cdots Se_3^{II}$	165.28(5)
$Se_2-Se_3\cdots Se_7^{III}$	168.49(6)	$Se_5-Se_7\cdots Se_6^{I}$	164.33(5)
$Se_5-Se_4\cdots Se_8^{II}$	170.35(5)	$Se_2-Se_8\cdots Se_1^{IV}$	167.53(5)
$Se_5-Se_6\cdots Se_7^{IV}$	170.84(5)	$Se_5-Se_8\cdots Se_4^{III}$	167.97(5)
$Se_5-Se_7\cdots Se_3^{II}$	98.79(5)	$Se_2-Se_8\cdots Se_4^{III}$	99.32(5)
$Se_2-Se_7\cdots Se_6^{I}$	100.84(5)	$Se_5-Se_8\cdots Se_1^{II}$	100.73(5)
$Se_6^{I}\cdots Se_7\cdots Se_3^{II}$	65.63(5)	$Se_4^{III}\cdots Se_8\cdots Se_1^{II}$	68.36(5)

Table 4. Bond lengths and short distances (Å) and angles (°) out of th plane of the eight selenium unit. Standard deviations in parentheses.

Se_1-C_1	1.828(13)	$\angle Se_1-C_1-N_1$	177.3(9)
C_1-N_1	1.149(17)	$\angle Se_2-Se_1-C_1$	95.2(4)
Se_3-C_3	1.806(13)	$\angle Se_3-C_3-N_3$	177.6(9)
C_3-N_3	1.120(17)	$\angle Se_2-Se_3-C_3$	95.0(5)
Se_4-C_4	1.838(13)	$\angle Se_4-C_4-N_4$	179.2(8)
C_4-N_4	1.136(17)	$\angle Se_5-Se_4-C_4$	94.5(4)
Se_6-C_6	1.829(13)	$\angle Se_6-C_6-N_6$	179.4(10)
C_6-N_6	1.145(17)	$\angle Se_5-Se_6-C_6$	94.7(4)
Se_7-C_7	1.799(12)	$\angle Se_7-C_7-N_7$	178.3(9)
C_7-N_7	1.149(14)	$\angle Se_2-Se_7-C_7$	83.7(4)
		$\angle Se_5-Se_7-C_7$	81.2(4)
Se_8-C_8	1.812(12)	$\angle Se_8-C_8-N_8$	178.1(8)
C_8-N_8	1.145(16)	$\angle Se_2-Se_8-C_8$	90.2(4)
		$\angle Se_5-Se_8-C_8$	97.0(4)
$Se_2\cdots Se_1^V$	3.369(2)	$\angle Se_1-Se_2\cdots Se_1^V$	92.09(5)
		$\angle Se_3-Se_2\cdots Se_1^V$	75.31(5)
		$\angle Se_7-Se_2\cdots Se_1^V$	102.10(5)
		$\angle Se_8-Se_2\cdots Se_1^V$	97.02(5)
$Se_5\cdots Se_4^{VI}$	3.452(2)	$\angle Se_4-Se_5\cdots Se_4^{VI}$	102.31(5)
		$\angle Se_6-Se_5\cdots Se_4^{VI}$	100.73(6)
		$\angle Se_7-Se_5\cdots Se_4^{VI}$	74.32(5)
		$\angle Se_8-Se_5\cdots Se_4^{VI}$	72.52(5)
$Se_1\cdots Se_2^V$	3.369(2)	$\angle Se_2-Se_1\cdots Se_2^V$	87.91(5)
		$\angle C_1-Se_1\cdots Se_2^V$	172.3(4)
$Se_4\cdots Se_5^{VI}$	3.452(2)	$\angle Se_5-Se_4\cdots Se_5^{VI}$	77.69(5)
		$\angle C_4-Se_4\cdots Se_5^{VI}$	170.7(4)
$Se_8\cdots Se_8^{VII}$	3.724(2)	$\angle Se_2-Se_8\cdots Se_8^{VII}$	84.41(5)
		$\angle Se_5-Se_8\cdots Se_8^{VII}$	110.17(5)
		$\angle C_8-Se_8\cdots Se_8^{VII}$	164.6(4)

Table 5. A comparison of the coordinates of the selenium atoms in the crystal structure of rubidium seleniumtriselenocyanate hemihydrate with the selenium coordinates in the corresponding potassium salt.[a]

	KSe(SeCN)₃·½H₂O			RbSe(SeCN)₃·½H₂O		
	X	Y	Z	X	Y	Z
Se_1	0.176	1.736	−1.164	0.278	1.764	−1.069
Se_2	0.287	1.572	1.251	0.359	1.543	1.340
Se_3	−2.124	1.315	1.218	−2.048	1.352	1.301
Se_4	6.239	1.575	4.923	6.362	1.492	5.028
Se_5	3.830	1.676	4.924	3.980	1.682	5.049
Se_6	3.928	1.706	7.335	4.022	1.602	7.445
Se_7	3.426	1.553	1.669	3.523	1.627	1.728
Se_8	0.615	1.704	4.537	0.684	1.736	4.707

[a] The reference coordinate system is a monoclinic system where the a and c axes of the triclinic cells are unchanged and the b axis is at right angle to the a and c axes. The coordinates of the monoclinic system, X, Y, Z in Å, relative to the triclinic cells, are given by:

$X = ax + by(\cos \gamma - \cos \alpha \cos \beta)/\sin^2 \beta$

$Y = by(1 - \cos^2 \alpha - \cos^2 \beta - \cos^2 \gamma + 2\cos \alpha \cos \beta \cos \gamma)^{\frac{1}{2}}/\sin \beta$

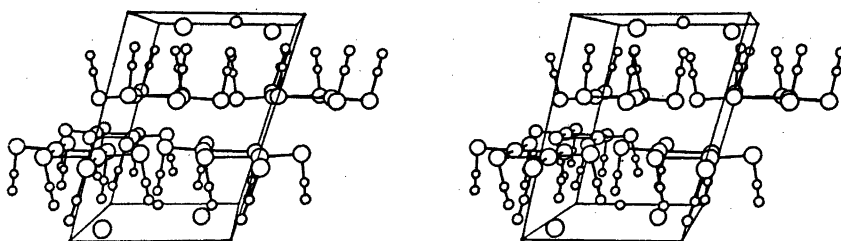$Z = cz + by(\cos \alpha - \cos \beta \cos \gamma)/\sin^2 \beta$

Fig. 2. A stereoscopic pair of drawings of $RbSe(SeCN)_3 \cdot \frac{1}{2}H_2O$. The cell drawn is along the *a* and *c* axes from 0 to 1 and along the *b* axis from $-\frac{1}{2}$ to $\frac{1}{2}$.

tainties, found to be a little longer than the others. The lengthening of the bonds was explained by the close $Se \cdots Se$ contacts between single layers, *trans* to the $Se-C$ bonds. The present structure has about the same $Se \cdots Se$ contacts between single layers, but the $Se-C$ bond lengthening is not observed.

A comparison of the crystal structure of the dimerized seleniumtriselenocyanate ion as it occurs in the potassium salt, with the structure of selenium diselenocyanate has been done earlier.[3] In the structure of selenium diselenocyanate [7,8] the $Se-Se$ bond lengths are 2.33 Å and the $Se-Se-Se$ angle is 103 °. Two short

$Se \cdots N$ contacts, 3.16 Å, occur in directions indicating a square planar coordination at the central selenium atom. The dimerized seleniumtriselenocyanate as it occurs in the potassium and rubidium salts, can be looked upon as built up of two selenium diselenocyanate molecules bridged together through the selenium atoms of two selenocyanate ions. The $Se-Se$ bonds of the $Se(SeCN)_2$ parts of the anion are approached by the selenium atoms of the selenocyanate ions, resulting in square planar coordinations at the central selenium atoms. In the present structure, the sums of the two $Se-Se$ bond lengths in the $Se(SeCN)_2$ parts of the

Table 6. Distances from the rubidium ions and from the water molecule. Bond lengths (Å) and angles (°). Standard deviations are given in parentheses.

$Rb_1 \cdots N_4$	3.224(11)	$\angle N_4 \cdots Rb_1 \cdots N_6$	66.5(3)
$Rb_1 \cdots N_6$	3.233(11)	$\angle N_4 \cdots Rb_1 \cdots N_7$	95.0(3)
$Rb_1 \cdots N_7$	3.080(11)	$\angle N_4 \cdots Rb_1 \cdots N_8$	129.3(3)
$Rb_1 \cdots N_8$	3.187(12)	$\angle N_6 \cdots Rb_1 \cdots N_7$	127.7(3)
$Rb_1 \cdots N_1^{IX}$	3.076(9)	$\angle N_6 \cdots Rb_1 \cdots N_8$	86.7(3)
$Rb_1 \cdots N_4^{VIII}$	3.101(13)	$\angle N_7 \cdots Rb_1 \cdots N_8$	67.6(3)
$Rb_1 \cdots N_8^{XI}$	3.087(11)	$\angle N_1^{IX} \cdots Rb_1 \cdots N_4^{VIII}$	80.8(3)
$Rb_1 \cdots O^{VIII}$	2.977(11)	$\angle N_1^{IX} \cdots Rb_1 \cdots N_8^{XI}$	70.9(3)
		$\angle N_4^{VIII} \cdots Rb \cdots N_8^{XI}$	71.5(3)
$Rb_2 \cdots N_1^{II}$	3.298(12)	$\angle N_1^{II} \cdots Rb_2 \cdots N_3^{II}$	103.7(3)
$Rb_2 \cdots N_3^{II}$	3.146(14)	$\angle N_1^{II} \cdots Rb_2 \cdots N_7^{II}$	62.3(3)
$Rb_2 \cdots N_7^{II}$	3.132(10)	$\angle N_1^{II} \cdots Rb_2 \cdots N_8^{II}$	124.0(3)
$Rb_2 \cdots N_8^{II}$	3.019(10)	$\angle N_3^{II} \cdots Rb_2 \cdots N_7^{II}$	122.6(3)
$Rb_2 \cdots N_1^{X}$	3.277(12)	$\angle N_3^{II} \cdots Rb_2 \cdots N_8^{II}$	80.2(3)
$Rb_2 \cdots N_8^{VIII}$	3.061(11)	$\angle N_7^{II} \cdots Rb_2 \cdots N_8^{II}$	69.1(3)
$Rb_2 \cdots N_7^{X}$	3.030(10)	$\angle N_1^{X} \cdots Rb_2 \cdots N_8^{VIII}$	65.5(3)
$Rb_2 \cdots O'$	2.907(9)	$\angle N_1^{X} \cdots Rb_2 \cdots N_7^{X}$	63.5(3)
		$\angle N_8^{VIII} \cdots Rb_2 \cdots N_7^{X}$	80.8(3)
$O \cdots N_3^{XI}$	2.916(15)	$\angle N_3^{XI} \cdots O \cdots N_6$	93.0(5)
$O \cdots N_6$	3.114(18)	$\angle N_3^{XI} \cdots O \cdots Rb_1^{VIII}$	105.3(4)
$O \cdots Rb_1^{VIII}$	2.977(11)	$\angle N_3^{XI} \cdots O \cdots Rb_2^{IV}$	95.0(4)
$O \cdots Rb_2^{IV}$	2.907(9)	$\angle N_6 \cdots O \cdots Rb_1^{VIII}$	123.0(4)
		$\angle N_6 \cdots O \cdots Rb_2^{IV}$	127.0(4)
		$\angle Rb_1^{VIII} \cdots O \cdots Rb_2^{VI}$	104.8(3)

anion are 4.784 and 4.815 Å, and the corresponding angles are 99.32 and 97.55 °, respectively. In the structure of the potassium salt, the sums are 4.806 and 4.822 Å and the angles are 98.41 and 97.60 °. From the three crystal structures it seems that an increase of $0.02 - 0.03$ Å in the sum of the two $Se-Se$ bond lengths corresponds to a decrease of one degree in the $Se-Se-Se$ angle.

Crystal packing. The rubidium ions and the water molecule lie at y approximately $\frac{1}{2}$, between double layers of selenium atoms around $y = 0$ and 1. The closest contacts of the potassium ions and of the water molecule are listed in Table 6. The water molecule is surrounded by two rubidium ions, at distances of 2.907 and 2.977 Å, and by two nitrogen atoms at distances of 2.916 and 3.114 Å. The latter contacts probably involve weak $O-H\cdots N$ hydrogen bonds. The $N\cdots O\cdots N$ angle is 93.3 °. The arrangement around the oxygen atom is approximately tetrahedral, the $Rb\cdots O\cdots Rb$ angle is 104.8 ° and the $N\cdots O\cdots Rb$ angles are in the range 95.0 to 127.0 °.

Each of the rubidium ions is surrounded by seven nitrogen atoms and one oxygen atom, with three of the nitrogen atoms in a plane above and four nitrogen atoms approximately in a plane below the rubidium ion; both planes are approximately parallel to the ac plane. The oxygen atom lies between the two planes. The $Rb\cdots N$ distances are in the range 3.020 Å to 3.298 Å, and the $Rb\cdots O$ distances are 2.907 and 2.977 Å.

The $Rb\cdots O$ distances are in the normal range (*cf.* Ref. 6, p. 259), and so are the $Rb\cdots N$ distances, the sum of the $Rb\cdots N$ ionic radii being 3.19 Å.[7]

REFERENCES

1. Verneuil, A. *Ann. Chim. Phys.* [6] *9* (1886) 289.
2. Hauge, S. *Acta Chem. Scand. 25* (1971) 3081.
3. Hauge, S. *Acta Chem. Scand. A 29* (1975) 771.
4. Coppens, P., Leiserowitz, L. and Rabinovich, D. *Acta Crystallogr. 18* (1965) 1035.
5. Hauge, S. *Acta Chem. Scand. A 29* (1975) 225.
6. Åse, K. *Acta Chem. Scand. 25* (1971) 838.
7. Aksnes, O. and Foss, O. *Acta Chem. Scand. 8* (1954) 1787.
8. McDonald, W. S. and Pettit, L. D. *J. Chem. Soc. A.* (1970) 2044.
9. *International Tables for X-Ray Crystallography*, Kynoch Press, Birmingham 1962, Vol III.
10. Pauling, L. *The Nature of the Chemical Bond.* 3rd. Ed., Cornell University Press, Ithaca, New York 1960.