base at the bottom. If the table rocks through an angle θ, the height of its centre of gravity alters by $\alpha\theta^2+\beta\theta^4+\ldots$ and it is easy to calculate that α becomes zero when the centre of gravity is one-third of the length of the spring below the upper clamp. It is convenient to place the centre of gravity near this point, finding a position such that the restoring forces of gravity and of the laminar supports combine to make the table only just stable in the central position. The main controlling force is then that of the torsion wire. The advantages of the lamina suspension are its freedom from friction, its high mechanical strength, and its rigidity in the axial direction which enables the torsion wire to be kept taut. This method is far superior to knife edges for a rocking arrangement such as the present. Setting by means of the spirit level is rapid and accurate. I am indebted to the head of the workshop, Mr Fuller, for this and many other suggestions. The setting of a dial is made positive by a small spring which presses into slits around the rim (to be seen in Fig. 3), so that the dial settles into place exactly opposite one of the hundred divisions. Two knurled segments of perspex, seen on either side in Fig. 2, make it easy to grasp the dial whatever its orientation. The weights are of brass, and the rest of the apparatus of aluminium, the table being made of duralumin 2 mm. in thickness. The torsion control is of steel piano wire 18 in. in length and 0·36 in. in diameter (20 s.w.g.). A half turn in either direction balances the moments of six carbon atoms. It would be a simple matter to extend the scale by balancer weights set at definite points on the table when required for a very large value of F, but in practice this has not been found necessary. The lamina supports of the table are of steel tape 1·2 in. × 0·375 in., and 0·15 in. thick. The construction of the calculator required some two to three weeks of workshop time.

I wish to express my warm thanks to Mr Fuller, and to Mr Collins who constructed the apparatus.

Reference

Vand, V. (1950). *T. Sci. Instrum.* **27**, 257.

Acta Cryst. (1952). **5**, 475

The Structure of Rubidium Trioxalatochromate (III), $Rb_3[Cr(C_2O_4)_3].xH_2O$, and its Relation to the Corresponding Potassium and Ammonium Structures

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By comparing Weissenberg photographs, Harker sections and Patterson projections it is shown that the compound $Rb_3[Cr(C_2O_4)_3].xH_2O$ and the corresponding potassium compound are isotype. The corresponding ammonium compound has a different structure. It is shown that this difference in structure is not due to the different sizes of the cations, but that it is probably due to the ability of an ammonium ion to form tetrahedrally directed hydrogen bonds.

Introduction

In two recent communications (van Niekerk & Schoening, 1952*a*, *b*) the crystal structures of potassium and ammonium trioxalatochromate (III) have been described. In the latter publication the two different schemes, according to which the complex ions and the cations are arranged in the two structures, were discussed. The question whether this difference in arrangement was due to (1) the difference in size of the cations, or (2) the ability of an ammonium ion to form hydrogen bonds, remained unanswered. To get further information as regards the first point it was decided to investigate the structures of the corresponding rubidium and cesium compounds. Goldschmidt and Pauling give the following data for the effective ionic radii of these ions:

	K^+	NH_4^+	Rb^+	Cs^+
Goldschmidt	1·33 Å	1·43 Å	1·49 Å	1·65 Å
Pauling	1·33	1·48	1·48	1·69

Although it has not been possible so far to crystallize the cesium compound, a structural analysis of the rubidium compound, as described below, seems to give a definite answer to the above question.

Preparation and crystal data

Crystals of $Rb_3[Cr(C_2O_4)_3].xH_2O$ were prepared following the method described by Werner (1912). The crystals are monoclinic and dark blue in colour. They generally crystallize as plates with well developed {010} and {021} faces round the [100] zone. In this respect they resemble the corresponding ammonium rather than the potassium compound.

From oscillation and Weissenberg photographs, using Cu $K\alpha$ and Mo $K\alpha$ radiation, the following unit-cell dimensions were obtained:

$$a=7{\cdot}81,\ b=19{\cdot}60,\ c=10{\cdot}40\ \text{Å},\ \beta=108^\circ\ 0'\ .$$

Assuming three molecules of water of crystallization in a formula unit, the calculated density is 2·75 g.cm.$^{-3}$. The ratios 1:1·0058:1·0315 give the respective relative volumes of corresponding unit cells for the potassium, rubidium and ammonium compounds.

Systematic extinctions occur only for $h0l$ with l odd and for $0k0$ with k odd. The space group is therefore $P2_1/c$. In this respect the compound resembles the corresponding potassium compound. The following data, used for a Harker section, show that the crystals very nearly have the pseudo space group $C2/c$. Fifty-eight spectra, contributing only 3·5% of the total intensity measured for 421 general hkl spectra, do not conform to the condition hkl present only with $h+k$ even. A similar condition was found for the corresponding potassium compound.

The crystal structure

Weissenberg photographs taken about the a axis were compared with corresponding photographs of the potassium compound. Except for small expected discrepancies in intensity, since rubidium now replaced potassium, the general appearance of the photographs was almost identical. From this it was inferred that the $b\,c$ projections of the two structures must be similar. This is confirmed by referring to Fig. 1, which shows the Patterson projections of the two compounds on the $b\,c$ plane.

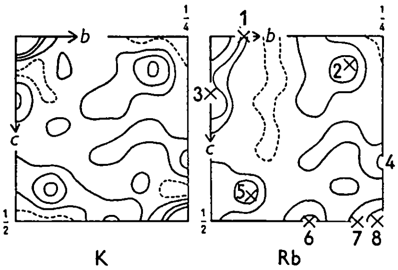

Fig. 1. Comparison of $P(0, u, w)$ for the potassium (K) and rubidium (Rb) compounds. Contours of each projection are drawn at arbitrary intervals; the lowest contour is a broken line. The crosses mark the positions of heavy-atom peaks as obtained from the final potassium structure. 1×: Cr–K_2; 2×: Cr–K_1, $\overline{K}_1$; 3×: K_1–$\overline{K}_1$; 4: frequently occurring O–O distance; 5×: Cr–K_1, $\overline{K}_1$; 6×: $\overline{K}_1$–$\overline{K}_1$; 7×: Cr–K_2; 8×: Cr–Cr.

There remained then to determine the arrangement of the complex ions and the cations in the a direction. There are two possible schemes of arrangement, one of these leading to the potassium and the other to the ammonium type of structure. The relation between these two schemes, involving a 180° rotation of complex ions and cations about the central chromium atom, was previously described in the ammonium publication where it was also outlined how, in the case of heavy cations, a Harker section would decide between the two possible schemes of arrangement. In Fig. 2 the Harker sections $P(0, v, \frac{1}{2}c)$ for the rubidium and the potassium compounds are shown. From the similarity of these sections, and the similarity of the $b\,c$ projections described above, it is concluded that the two structures are isotype. This is further confirmed by the fact that it was possible to obtain single mixed crystals of the two compounds, such crystals showing a doubling of the a spacing.

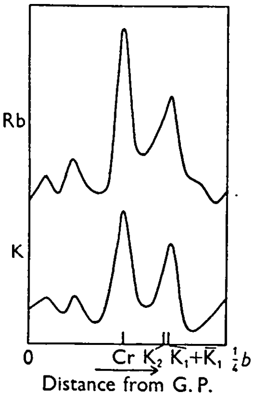

Fig. 2. Comparison of $P(0, v, \frac{1}{2}c)$ for the rubidium (Rb) and potassium (K) compounds. Sections are drawn to arbitrary intensity scales. Positions of the Cr and K atoms, as determined from the final potassium structure, are marked.

Conclusion

From this investigation, and accepting the ionic radii given by Goldschmidt and Pauling to be correct, it can be concluded that the structural difference between the ammonium compound and the two structurally similar potassium and rubidium compounds is not due to the size difference of the cations. It is tempting to associate this difference with the ability of an ammonium ion to form tetrahedrally directed hydrogen bonds, an arrangement found in this and many other ammonium compounds. This would lead to the ammonium structure being the more open one, as was indeed found to be the case.

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References

Niekerk, J. N. van & Schoening, F. R. L. (1952*a*). *Acta Cryst.* **5**, 196.

Niekerk, J. N. van & Schoening, F. R. L. (1952*b*). *Acta Cryst.* **5**, 499.

Werner, A. (1912). *Ber. dtsch. chem. Ges.* **45**, 3061.