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Structure of Rubidium Hydrogen (+)-Tartrate

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Abstract. [$RbH(C_4H_4O_6)$], $M_r = 234{\cdot}55$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}9233\,(8)$, $b = 10{\cdot}9883\,(9)$, $c = 7{\cdot}6527\,(22)$ Å, $V = 666{\cdot}3\,(2)$ Å^3, $Z = 4$, $D_m = 2{\cdot}29$, $D_x = 2{\cdot}338\,(1)$ g cm^{-3}, Mo $K\alpha$, $\lambda(\alpha_1) = 0{\cdot}70930$ Å, $\mu = 72{\cdot}1$ cm^{-1}, $F(000) = 456$, $T = 299$ K, $R = 0{\cdot}014$, $wR = 0{\cdot}015$ for 1100 independent reflections with $I > \sigma(I)$. The structure is nearly the same as that of caesium hydrogen tartrate except for cation–oxygen distances shorter by 0·15 Å on average. The anomalous scattering term f'' for rubidium measured at 0·71 Å is 2·90 (4).

Experimental. A mixture of RbCl, (+)-tartaric acid and water was heated and allowed to cool. Two crystals were studied. The first, with diameters 0·2 to 0·3 mm, was described by 17 faces. Absorption corrections did not agree well with intensities measured at different azimuths, and R could not be reduced below 0·042 for 978 independent reflections. Another crystal 0·021 × 0·060 × 0·37 mm (seven faces, elongated on **c**) was glued to a glass fiber and mounted on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. Cell dimensions were derived from 19 reflections in the range $16{\cdot}4 < \theta < 27{\cdot}7°$. Intensities were measured (θ–2θ scan) for θ up to 25° for h −9 to 9, k −12 to 0, l −9 to 9. Of 2443 reflections permitted by the space group, 196 were rejected as too weak to measure ($I < \sigma$). After analytical correction for absorption ($1{\cdot}16 < A < 1{\cdot}51$) reflections which were equivalent in point group 222 were averaged giving 1102 unique ones with $F^2 > \sigma(F^2)$; $R_{\rm int} = 0{\cdot}019$. Measurements of standard reflections (400, $0\bar{6}0$, $00\bar{6}$) every 7200 s of X-ray exposure were constant within about 2%; no correction was made. Refinement by least squares (on F) with weights $w = [\sigma(F)]^{-2}$, derived from $\sigma(F^2) = \{[\sigma_{\rm cs}(F^2)]^2 + (0{\cdot}017F^2)^2\}^{1/2}$, was started with parameters for the caesium isomorph (Templeton & Templeton, 1978). Anisotropic thermal parameters were assigned to 11 atoms and isotropic ones to five hydrogen atoms. The dispersion correction f'' for rubidium was included as a variable. Two reflections were rejected because of poor agreement with calculations: 130, the strongest one, and 020, another low-angle one. An empirical isotropic correction for extinction increased F by 1% for the strongest remaining reflection. The maximum final shift was $0{\cdot}005\,\sigma$; extremes of a ΔF synthesis were +0·19 and −0·21 e Å^{-3}; $S = 1{\cdot}12$. Atomic scattering factors, including dispersion terms, for Rb$^+$, C, O (for O1 to O4), O$^{1/2-}$ (for O5, O6) and H were taken from *International Tables for X-ray Crystallography* (1974). Calculations were made with unpublished local programs. Atomic parameters are listed in Table 1 and interatomic distances and angles in Table 2, with atoms numbered as in Templeton & Templeton (1978).[*] Positions for non-hydrogen atoms found in the unsatisfactory first experiment differ by 0·02 Å or less. The measured density is quoted from van Bommel & Bijvoet (1958).

Related literature. Potassium, rubidium, caesium, ammonium and thallium(I) hydrogen (+)-tartrates are all isomorphous (Groth, 1910). Crystals of the ammonium, potassium and rubidium salts were studied with X-rays by van Bommel & Bijvoet (1958). They refined the parameters (including hydrogen-atom coordinates) for the ammonium salt and used data from the rubidium salt to verify the absolute configuration of the tartrate ion. The parameters for the caesium compound were determined by Templeton & Templeton (1978) and used to measure anomalous scattering of synchrotron radiation by caesium near the L absorption edges

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[*] Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51554 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates and isotropic thermal parameters* (Å²) *with e.s.d.'s in parentheses*

$$B_{eq} = \sum B_{ii}/3.$$

	x	y	z	B or B_{eq}
Rb	0·33047 (3)	0·03378 (2)	1·16264 (3)	1·71
O1	0·62081 (27)	−0·13513 (18)	1·04696 (24)	2·07
O2	0·70363 (23)	−0·32806 (15)	1·01999 (23)	2·11
O3	0·57468 (23)	−0·34298 (15)	0·69143 (23)	1·52
O4	0·86066 (21)	−0·17134 (19)	0·68467 (26)	1·67
O5	0·47342 (20)	−0·10666 (16)	0·46010 (21)	1·66
O6	0·72967 (22)	−0·15314 (17)	0·36071 (23)	1·97
C1	0·64073 (28)	−0·23754 (23)	0·96042 (29)	1·32
C2	0·5771 (3)	−0·22683 (22)	0·7736 (3)	1·26
C3	0·69001 (28)	−0·13951 (20)	0·6712 (3)	1·18
C4	0·62516 (27)	−0·13157 (20)	0·4824 (3)	1·20
H1	0·458 (3)	−0·1916 (21)	0·773 (3)	0·7 (5)
H2	0·679 (3)	−0·0728 (23)	0·7215 (29)	0·9 (5)
H3	0·476 (4)	−0·3522 (23)	0·662 (4)	1·5 (6)
H4	0·870 (3)	−0·2259 (21)	0·654 (3)	0·1 (6)
H5	0·646 (4)	−0·138 (3)	1·124 (4)	2·2 (8)

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

C1—O1	1·315 (3)	O1—C1—O2	124·5 (2)
C1—O2	1·202 (3)	O1—C1—C2	111·6 (2)
C1—C2	1·521 (3)	O2—C1—C2	123·9 (2)
C2—O3	1·423 (3)	C1—C2—C3	109·7 (2)
C2—C3	1·528 (3)	C1—C2—O3	110·5 (2)
C3—O4	1·400 (3)	O3—C2—C3	110·2 (2)
C3—C4	1·536 (3)	C2—C3—C4	108·8 (2)
C4—O5	1·245 (3)	C2—C3—O4	111·8 (2)
C4—O6	1·268 (3)	O4—C3—C4	114·0 (2)
O1—O6^{i}	2·559 (3)*	C3—C4—O5	117·7 (2)
O3—O6^{ii}	2·763 (2)*	C3—C4—O6	117·5 (2)
O4—O5^{iii}	2·824 (3)*	O5—C4—O6	124·8 (2)
Rb—O2	2·841 (2)	Rb—O5	2·974 (2)
Rb—O2	2·877 (2)	Rb—O1	3·085 (2)
Rb—O4	2·881 (2)	Rb—O3	3·121 (2)
Rb—O5	2·973 (2)	Rb—O3	3·122 (2)

* Hydrogen bond; symmetry codes: (i) $x, y, 1+z$; (ii) $-\frac{1}{2}+x, -\frac{1}{2}-y, 1-z$; (iii) $\frac{1}{2}+x, -\frac{1}{2}-y, 1-z$.

(Templeton, Templeton, Phillips & Hodgson, 1980). Covalent-bond distances in the caesium and rubidium compounds are nearly identical, but the average distances from rubidium and caesium to the eight nearest oxygen neighbors are 2·984 and 3·131 Å. The purpose of the present study was to obtain parameters needed for similar measurements of rubidium anomalous scattering near its K absorption edge, to be described elsewhere. All of this work is in agreement about the absolute configuration. The value $f'' = 2{\cdot}973$ calculated by Cromer & Liberman (1970) for rubidium at Mo $K\alpha_1$ is close to that found here, 2·90 (4).

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N-3,4-Dichlorophenyl-3-methylbutanamide

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Abstract. $C_{11}H_{13}Cl_2NO$, $M_r = 246{\cdot}1$, triclinic, $P\bar{1}$, $a = 4{\cdot}818\,(1)$, $b = 10{\cdot}933\,(2)$, $c = 12{\cdot}309\,(3)$ Å, $\alpha = 105{\cdot}99\,(2)$, $\beta = 97{\cdot}86\,(2)$, $\gamma = 95{\cdot}28\,(2)°$, $V = 611{\cdot}6\,(5)$ Å³, $D_x = 1{\cdot}34$ Mg m^{-3}, $Z = 2$, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 4{\cdot}66$ mm^{-1}, $F(000) = 256$, $T = 293$ K. Final $R = 0{\cdot}067$ for 1551 observed $[F > 3\sigma(F_o)]$ reflections and 178 parameters. The phenyl and amido groups are planar and form a dihedral angle of 47·7 (5)°. This may be compared with values of 26·6 (6)° in *N*-3,4-dichlorophenyl-2-methylpropanamide [Precigoux, Busetta & Hospital (1976). *Acta Cryst.* B**32**, 943–945] and 17·1 (9)° in *N*-phenylacetamide [Brown (1966). *Acta Cryst.* **21**, 442–445]. The C_{ar}—N and N—C(=O) bond lengths are 1·420 (3) and 1·344 (4) Å respectively. The mean C—Cl bond length is 1·731 (3) Å. The crystal structure is stabilized

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