

Crystal structure of trirubidium citrate monohydrate from laboratory X-ray powder diffraction data and DFT comparison

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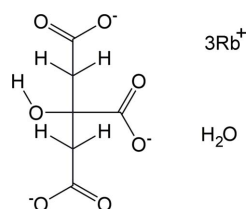
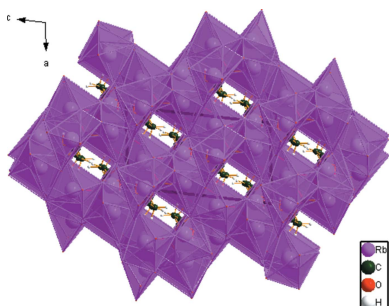
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The crystal structure of the title compound, $3\text{Rb}^+ \cdot \text{C}_6\text{H}_5\text{O}_7^{3-} \cdot \text{H}_2\text{O}$, has been solved and refined using laboratory X-ray powder diffraction data, and optimized using density functional techniques. The hydroxy group participates in an intramolecular hydrogen bond to the deprotonated central carboxylate group with graph-set motif $S(5)$. The water molecule acts as a hydrogen-bond donor to both terminal and central carboxylate O atoms. The three independent rubidium cations are seven-, six- and six-coordinate, with bond-valence sums of 0.84, 1.02, and 0.95, respectively. In the extended structure, their polyhedra share edges and corners to form a three-dimensional network. The hydrophobic methylene groups occupy channels along the b axis.

1. Chemical context

In the course of a systematic study of the crystal structures of Group 1 (alkali metal) citrate salts to understand the anion's conformational flexibility, deprotonation, coordination tendencies, and hydrogen bonding, we have determined several new crystal structures. Most of the new structures were solved using powder diffraction data (laboratory and/or synchrotron), but single crystals were used where available. The general trends and conclusions about the sixteen new compounds and twelve previously characterized structures are being reported separately (Rammohan & Kaduk, 2017*a*). Seven of the new structures – $\text{NaKHC}_6\text{H}_5\text{O}_7$, $\text{NaK}_2\text{C}_6\text{H}_5\text{O}_7$, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$, $\text{NaH}_2\text{C}_6\text{H}_5\text{O}_7$, $\text{Na}_2\text{HC}_6\text{H}_5\text{O}_7$, $\text{K}_3\text{C}_6\text{H}_5\text{O}_7$, and $\text{Rb}_2\text{HC}_6\text{H}_5\text{O}_7$ – have been published recently (Rammohan & Kaduk, 2016*a,b,c,d,f*, 2017*b*; Rammohan *et al.* (2016)), and two additional structures – $\text{KH}_2\text{C}_6\text{H}_5\text{O}_7$ and $\text{KH}_2\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})_2$ – have been communicated to the CSD (Kaduk & Stern, 2016*a,b*).



2. Structural commentary

The asymmetric unit of the title compound is shown in Fig. 1. The root-mean-square deviation of the non-hydrogen atoms in

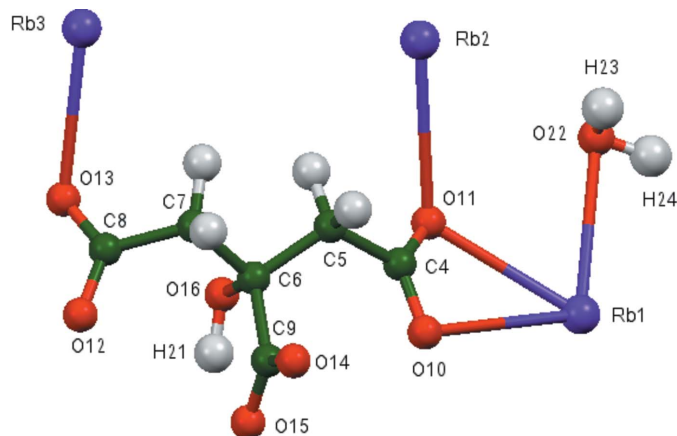


Figure 1
The asymmetric unit, with the atom numbering. The atoms are represented by 50% probability spheroids.

the Rietveld-refined and DFT-optimized structures is 0.127 Å (Fig. 2). The good agreement between the two structures is strong evidence that the experimental structure is correct (van de Streek & Neumann, 2014). This discussion uses the DFT-optimized structure. Most of the bond lengths, bond angles, and torsion angles fall within the normal ranges indicated by a *Mercury* Mogul geometry check (Macrae *et al.*, 2008). Only the O11–C4–C5–C6 torsion angle involving a terminal carboxylate group is flagged as unusual, but as shown in Rammohan & Kaduk (2017*a*) these torsion angles exhibit no real preference. The citrate anion occurs in the *trans,trans*-conformation, which is one of the two low-energy conformations of an isolated citrate trianion. The central carboxylate group and the hydroxyl group occur in the normal planar arrangement. The terminal carboxylate O13 atom and the hydroxyl group O16 atom chelate to Rb3. The terminal carboxylate O12 atom and the central carboxylate O15 atom

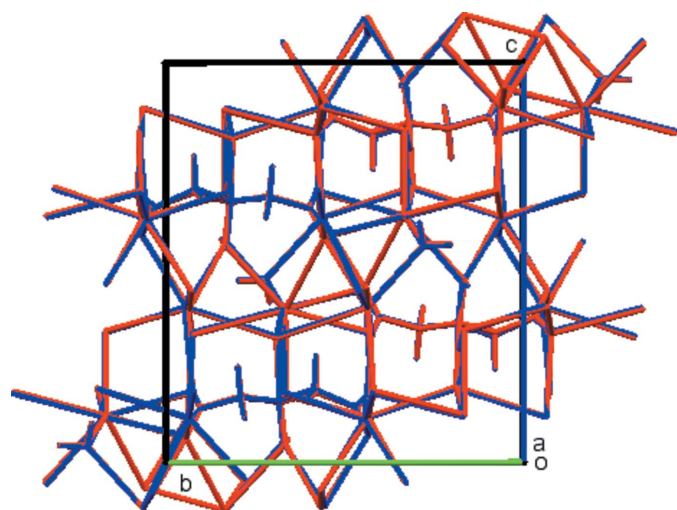


Figure 2
Comparison of the refined and optimized structures of trirubidium citrate monohydrate. The refined structure is in red, and the DFT-optimized structure is in blue.

Table 1
Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O16–H21···O15	0.984	1.838	2.552	126.9
O22–H23···O14 ⁱ	0.983	1.704	2.672	168.7
O22–H24···O13	0.984	1.707	2.683	170.8
C5–H17···O22	1.093	2.674	3.749	167.4

Symmetry code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$.

chelate to another Rb3 cation. The terminal carboxylate O12 and central carboxylate O14 chelate to Rb2, and the terminal O10 and central O14 chelate to a third Rb3 atom. The terminal carboxylate O14/O15 acts as a bidentate ligand to Rb1, and the terminal carboxylate O10/O11 chelates to another Rb1.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) morphology suggests that we might expect platy morphology for the title compound, with {011} as the principal faces. A 4th-order spherical harmonic texture model was included in the refinement. The texture index was 1.014, indicating that preferred orientation was negligible for this rotated flat-plate specimen.

3. Supramolecular features

The three independent Rb⁺ ions are 7-, 6- and 6-coordinate (upper threshold for Rb–O bond lengths = 3.40 Å), with bond-valence sums of 0.84, 1.02, and 0.95, respectively. These polyhedra share edges and corners to form a three-dimensional network (Fig. 3). Hydrogen bonds (Table 1) between the water molecules and the citrate anions result in chains propagating along the *b*-axis direction. The hydroxyl group participates in an intramolecular hydrogen bond to the deprotonated central carboxylate group with graph-set motif *S*(5). The water molecule acts as a hydrogen-bond donor to both the terminal carboxylate atom O13 and the central

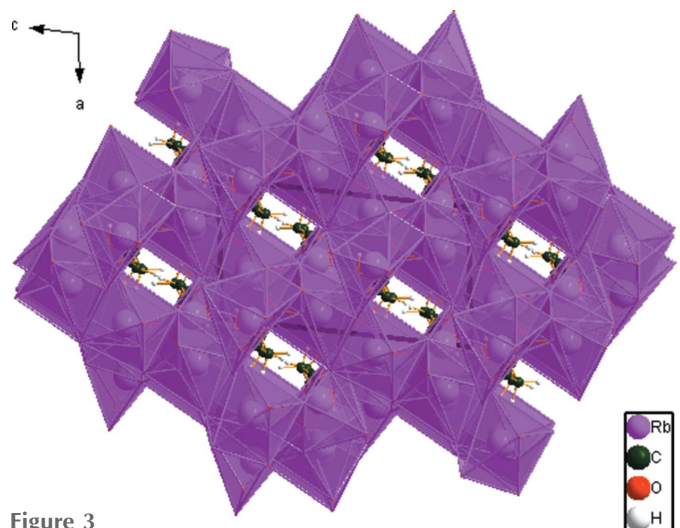
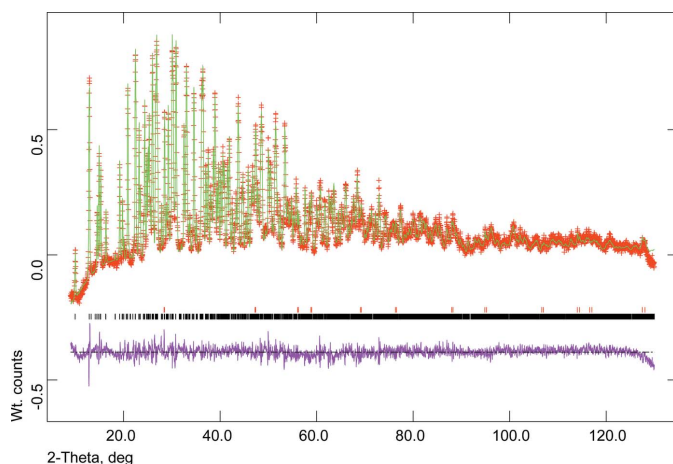


Figure 3
Crystal structure of trirubidium citrate monohydrate, viewed down the *b* axis. **outline of the unit cell needs to be added**


Figure 4

Rietveld plot for the refinement of trirubidium citrate monohydrate. The vertical scale is not the raw counts but the counts multiplied by the least squares weights. This plot emphasizes the fit of the weaker peaks. The red crosses represent the observed data points, and the green line is the calculated pattern. The magenta curve is the difference pattern, plotted at the same scale as the other patterns. The row of black tick marks indicates the reflection positions, and the red tick marks indicate the Si internal standard peak positions.

carboxylate atom O14. The Mulliken overlap populations indicate, by the correlation in Rammohan & Kaduk (2017a), that these hydrogen bonds account for $41.6 \text{ kcal mol}^{-1}$ of crystal energy. A C—H...O hydrogen bond also apparently contributes to the crystal energy. The hydrophobic methylene groups occupy channels along the *b*-axis. This compound is isostructural to $\text{K}_3\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})$ (Carrell *et al.*, 1987; CSD Refcode ZZZHVI01).

4. Database survey

Details of the comprehensive literature search for citrate structures are presented in Rammohan & Kaduk (2017a). A reduced cell search of the cell of trirubidium citrate monohydrate in the Cambridge Structural Database (Groom *et al.*, 2016) (increasing the default tolerance from 1.5 to 2.0%) yielded 228 hits, but combining the cell search with a citrate fragment yielded Love & Patterson (1960, CSD Refcode ZZZHZC), but no coordinates were reported for this phase. Increasing the tolerance on the cell to 5% yielded $\text{K}_3\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})$ (Burns & Iball, 1954, CSD Refcode ZZZHVI; Carrell *et al.*, 1987, CSD Refcodes ZZZHVI01 and ZZZHVI02).

5. Synthesis and crystallization

$\text{H}_3\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})$ (10.0 mmol, 2.0972 g) was dissolved in 10 ml deionized water. Rb_2CO_3 (15.0 mmol, 3.4659 g, Sigma-Aldrich) was added to the citric acid solution slowly with stirring. The resulting clear colourless solution was evaporated to dryness at ambient conditions to yield a white powder.

Table 2

Experimental details.

	Powder data
Crystal data	
Chemical formula	$3\text{Rb}^+\cdot\text{C}_6\text{H}_5\text{O}_7^{3-}\cdot\text{H}_2\text{O}$
M_r	463.52
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	300
a, b, c (Å)	7.44769 (10), 11.87554 (16), 13.41675 (18)
β (°)	97.8820 (9)
V (Å ³)	1175.44 (4)
Z	4
Radiation type	$K\alpha_1, K\alpha_2, \lambda = 1.540629, 1.544451 \text{ Å}$
Specimen shape, size (mm)	Flat sheet, 24×24
Data collection	
Diffractometer	IIT Bruker D2 Phaser
Specimen mounting	Si zero-background cell
Data collection mode	Reflection
Scan method	Step
2θ values (°)	$2\theta_{\min} = 5.042$ $2\theta_{\max} = 130.045$ $2\theta_{\text{step}} = 0.020$
Refinement	
R factors and goodness of fit	$R_p = 0.015$, $R_{\text{wp}} = 0.019$, $R_{\text{exp}} = 0.007$, $R(F^2) = 0.061$, $\chi^2 = 8.352$
No. of parameters	88
No. of restraints	47

The same symmetry and lattice parameters were used for the DFT calculation. Computer programs: *DIFFRAC.Measurement* (Bruker, 2009), (*GSAS*, Larson & Von Dreele, 2004), *DIAMOND* (Crystal Impact, 2015) and *publCIF* (Westrip, 2010).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The specimen was blended with a NIST SRM 640 Si internal standard ($a = 5.43105 \text{ Å}$). The powder pattern (Fig. 4) was indexed using *Jade 9.4* (MDI, 2012), which yielded a primitive monoclinic cell having $a = 7.44769$ (10), $b = 11.87554$ (16), $c = 13.41675$ (18) Å, $\beta = 97.8820$ (9)°, $V = 1175.44$ (3) Å³, and $Z = 4$. The suggested space group was $P2_1/n$, which was confirmed by successful solution and refinement. Three intense peaks from a structure solution using charge flipping as implemented in *Jana2006* (Petříček *et al.*, 2014) were used to carry out a Le Bail fit in *GSAS* (Larson & Von Dreele, 2004). The resulting peak list was imported into *Endeavour 1.7b* (Putz *et al.*, 1999), which was used to solve the structure with a citrate anion and 3 Rb atoms as fragments. A significant peak in a difference Fourier map in *GSAS* corresponded to the oxygen atom of a water molecule, indicating that the compound was a monohydrate.

Pseudo-Voigt profile coefficients were as parameterized in Thompson *et al.* (1987) with profile coefficients for Simpson's rule integration of the pseudo-Voigt function according to Howard (1982). The asymmetry correction of Finger *et al.* (1994) was applied, and microstrain broadening by Stephens (1999). The structure was refined by the Rietveld method using *GSAS/EXPGUI* (Larson & Von Dreele, 2004; Toby, 2001). All C—C and C—O bond lengths were restrained, as were all bond angles. The hydrogen atoms were included at fixed positions, which were recalculated during the course of

the refinement using *Materials Studio* (Dassault Systemes, 2014). The U_{iso} values of the C and O atoms in the citrate anion were constrained to be equal, and the U_{iso} values of the hydrogen atoms were constrained to be 1.3 times those of the atoms to which they are attached.

7. DFT calculations

After the Rietveld refinement, a density functional geometry optimization (fixed experimental unit cell) was carried out using *CRYSTAL09* (Dovesi *et al.*, 2005). The basis sets for the C, H, and O atoms were those of Gatti *et al.* (1994), and the basis set for Rb was that of Schoenes *et al.* (2008). The calculation used 8 k -points and the B3LYP functional, and took about 72 h on a 2.4 GHz PC. The U_{iso} values from the Rietveld refinement were assigned to the optimized fractional coordinates.

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Crystal structure of trirubidium citrate monohydrate from laboratory X-ray powder diffraction data and DFT comparison

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Computing details

(RAMM010_phase_1)

Crystal data

$3\text{Rb}^+\cdot\text{C}_6\text{H}_5\text{O}_7^{3-}\cdot\text{H}_2\text{O}$

$M_r = 463.52$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 7.44769$ (10) Å

$b = 11.87554$ (16) Å

$c = 13.41675$ (18) Å

$\beta = 97.8820$ (9)°

$V = 1175.44$ (4) Å³

$Z = 4$

$D_x = 2.619$ Mg m⁻³

$T = 300$ K

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Rb1	0.0102 (2)	0.1660 (2)	0.11841 (17)	0.0456 (4)*
Rb2	0.1182 (3)	0.43646 (19)	0.38731 (17)	0.0456 (4)*
Rb3	0.3356 (2)	0.4349 (2)	0.11309 (16)	0.0456 (4)*
C4	0.886 (2)	0.9013 (13)	0.1375 (12)	0.033 (3)*
C5	0.8103 (19)	0.7886 (13)	0.1665 (13)	0.033 (3)*
C6	0.9144 (18)	0.6786 (13)	0.1510 (9)	0.033 (3)*
C7	0.794 (2)	0.5780 (14)	0.1757 (11)	0.033 (3)*
C8	0.847 (2)	0.4649 (15)	0.1373 (13)	0.033 (3)*
C9	1.1021 (17)	0.6830 (16)	0.2152 (9)	0.033 (3)*
O10	1.0404 (15)	0.9238 (11)	0.1753 (9)	0.0351 (13)*
O11	0.7895 (15)	0.9429 (11)	0.0622 (9)	0.0351 (13)*
O12	0.9890 (15)	0.4210 (10)	0.1791 (8)	0.0351 (13)*
O13	0.7342 (15)	0.4316 (11)	0.0691 (10)	0.0351 (13)*
O14	1.0988 (13)	0.6888 (12)	0.3119 (7)	0.0351 (13)*
O15	1.2398 (14)	0.6654 (9)	0.1693 (7)	0.0351 (13)*
O16	0.9233 (13)	0.6642 (11)	0.0459 (8)	0.0351 (13)*
H17	0.66841	0.77938	0.11192	0.042 (4)*
H18	0.76254	0.79781	0.23982	0.042 (4)*
H19	0.64492	0.60516	0.15820	0.042 (4)*
H20	0.80258	0.57869	0.26635	0.042 (4)*
H21	1.06826	0.65697	0.05902	0.0456 (16)*
O22	0.6164 (13)	0.2217 (9)	0.0449 (8)	0.040 (4)*

H23	0.54454	0.20759	0.09843	0.052 (5)*
H24	0.65210	0.29614	0.04430	0.052 (5)*

Geometric parameters (Å, °)

Rb1—O10 ⁱ	2.976 (12)	C8—O12	1.239 (12)
Rb1—O11 ⁱ	3.153 (13)	C8—O13	1.221 (13)
Rb1—O11 ⁱⁱ	3.281 (11)	C9—C6	1.539 (8)
Rb1—O12 ⁱⁱⁱ	3.146 (12)	C9—O14	1.302 (11)
Rb1—O13 ⁱⁱⁱ	3.773 (12)	C9—O15	1.285 (10)
Rb1—O14 ^{iv}	2.946 (10)	O10—Rb1 ^{viii}	2.976 (12)
Rb1—O15 ^{iv}	3.182 (10)	O10—Rb2 ^{ix}	2.789 (11)
Rb1—O16 ⁱⁱ	3.078 (11)	O10—Rb3 ^{ix}	2.866 (12)
Rb1—O22 ⁱⁱⁱ	3.036 (10)	O10—C4	1.222 (12)
Rb2—O10 ^{iv}	2.789 (11)	O11—Rb1 ^{viii}	3.153 (13)
Rb2—O11 ^v	3.201 (10)	O11—Rb1 ⁱⁱ	3.281 (11)
Rb2—O11 ^{vi}	2.892 (12)	O11—Rb2 ^x	3.201 (10)
Rb2—O12 ⁱⁱⁱ	2.833 (11)	O11—Rb2 ^{xi}	2.892 (12)
Rb2—O14 ⁱⁱⁱ	3.160 (13)	O11—C4	1.257 (12)
Rb2—O15 ^{iv}	3.504 (11)	O12—Rb1 ^{xii}	3.146 (12)
Rb2—O22 ^{vii}	2.830 (10)	O12—Rb2 ^{xii}	2.833 (11)
Rb3—O10 ^{iv}	2.866 (12)	O12—Rb3 ^{xii}	2.847 (11)
Rb3—O12 ⁱⁱⁱ	2.847 (11)	O12—C8	1.239 (12)
Rb3—O13	3.105 (11)	O13—Rb1 ^{xii}	3.773 (12)
Rb3—O13 ⁱⁱ	2.900 (13)	O13—Rb3	3.105 (11)
Rb3—O14 ^{iv}	3.108 (13)	O13—Rb3 ⁱⁱ	2.900 (13)
Rb3—O15 ⁱⁱⁱ	2.952 (10)	O13—C8	1.221 (13)
Rb3—O16 ⁱⁱ	2.920 (11)	O14—Rb1 ^{ix}	2.946 (10)
Rb3—O22	3.485 (11)	O14—Rb2 ^{xii}	3.160 (13)
C4—C5	1.522 (8)	O14—Rb3 ^{ix}	3.108 (13)
C4—O10	1.222 (12)	O14—C9	1.302 (11)
C4—O11	1.257 (12)	O15—Rb1 ^{ix}	3.182 (10)
C5—C4	1.522 (8)	O15—Rb2 ^{ix}	3.504 (11)
C5—C6	1.548 (8)	O15—Rb3 ^{xii}	2.952 (10)
C6—C5	1.548 (8)	O15—C9	1.285 (10)
C6—C7	1.555 (8)	O16—Rb1 ⁱⁱ	3.078 (11)
C6—C9	1.539 (8)	O16—Rb3 ⁱⁱ	2.920 (11)
C6—O16	1.431 (8)	O16—C6	1.431 (8)
C7—C6	1.555 (8)	O22—Rb1 ^{xii}	3.036 (10)
C7—C8	1.511 (8)	O22—Rb2 ^{xiii}	2.830 (10)
C8—C7	1.511 (8)	O22—Rb3	3.485 (11)
O10 ⁱ —Rb1—O11 ⁱ	43.0 (3)	O12 ⁱⁱⁱ —Rb2—O22 ^{xv}	131.2 (3)
O10 ⁱ —Rb1—O11 ⁱⁱ	77.4 (3)	O14 ⁱⁱⁱ —Rb2—O22 ^{xv}	149.8 (3)
O10 ⁱ —Rb1—O12 ⁱⁱⁱ	150.3 (3)	O10 ^{iv} —Rb3—O12 ⁱⁱⁱ	82.6 (3)
O10 ⁱ —Rb1—O14 ^{iv}	88.3 (4)	O10 ^{iv} —Rb3—O13	90.1 (4)
O10 ⁱ —Rb1—O15 ^{iv}	75.8 (3)	O10 ^{iv} —Rb3—O13 ⁱⁱ	148.9 (4)
O10 ⁱ —Rb1—O16 ⁱⁱ	143.4 (3)	O10 ^{iv} —Rb3—O14 ^{iv}	67.7 (3)

O10 ⁱ —Rb1—O22 ⁱⁱⁱ	109.2 (3)	O10 ^{iv} —Rb3—O15 ⁱⁱⁱ	81.2 (3)
O11 ⁱ —Rb1—O11 ⁱⁱ	76.5 (3)	O10 ^{iv} —Rb3—O16 ⁱⁱ	142.7 (4)
O11 ⁱ —Rb1—O12 ⁱⁱⁱ	145.8 (3)	O12 ⁱⁱⁱ —Rb3—O13	171.8 (4)
O11 ⁱ —Rb1—O14 ^{iv}	127.8 (4)	O12 ⁱⁱⁱ —Rb3—O13 ⁱⁱ	103.2 (3)
O11 ⁱ —Rb1—O15 ^{iv}	115.7 (3)	O12 ⁱⁱⁱ —Rb3—O14 ^{iv}	87.3 (3)
O11 ⁱ —Rb1—O16 ⁱⁱ	120.6 (3)	O12 ⁱⁱⁱ —Rb3—O15 ⁱⁱⁱ	73.3 (4)
O11 ⁱ —Rb1—O22 ⁱⁱⁱ	69.9 (3)	O12 ⁱⁱⁱ —Rb3—O16 ⁱⁱ	70.6 (3)
O11 ⁱⁱ —Rb1—O12 ⁱⁱⁱ	127.9 (3)	O13—Rb3—O13 ⁱⁱ	85.0 (4)
O11 ⁱⁱ —Rb1—O14 ^{iv}	75.0 (3)	O13—Rb3—O14 ^{iv}	86.4 (3)
O11 ⁱⁱ —Rb1—O15 ^{iv}	113.0 (3)	O13—Rb3—O15 ⁱⁱⁱ	109.3 (4)
O11 ⁱⁱ —Rb1—O16 ⁱⁱ	66.1 (3)	O13—Rb3—O16 ⁱⁱ	114.1 (3)
O11 ⁱⁱ —Rb1—O22 ⁱⁱⁱ	111.3 (3)	O13 ⁱⁱ —Rb3—O14 ^{iv}	142.1 (3)
O12 ⁱⁱⁱ —Rb1—O14 ^{iv}	84.9 (3)	O13 ⁱⁱ —Rb3—O15 ⁱⁱⁱ	71.8 (3)
O12 ⁱⁱⁱ —Rb1—O15 ^{iv}	79.3 (3)	O13 ⁱⁱ —Rb3—O16 ⁱⁱ	65.2 (4)
O12 ⁱⁱⁱ —Rb1—O16 ⁱⁱ	64.7 (3)	O14 ^{iv} —Rb3—O15 ⁱⁱⁱ	145.3 (3)
O12 ⁱⁱⁱ —Rb1—O22 ⁱⁱⁱ	78.1 (3)	O14 ^{iv} —Rb3—O16 ⁱⁱ	85.2 (3)
O14 ^{iv} —Rb1—O15 ^{iv}	44.3 (2)	O15 ⁱⁱⁱ —Rb3—O16 ⁱⁱ	113.7 (3)
O14 ^{iv} —Rb1—O16 ⁱⁱ	85.3 (3)	C5—C4—O10	116.5 (15)
O14 ^{iv} —Rb1—O22 ⁱⁱⁱ	162.1 (3)	C5—C4—O11	111.2 (15)
O15 ^{iv} —Rb1—O16 ⁱⁱ	120.7 (3)	O10—C4—O11	130.4 (16)
O15 ^{iv} —Rb1—O22 ⁱⁱⁱ	135.4 (3)	C4—C5—C6	119.9 (13)
O16 ⁱⁱ —Rb1—O22 ⁱⁱⁱ	82.4 (3)	C5—C6—C7	107.8 (11)
O10 ^{iv} —Rb2—O11 ^v	174.5 (4)	C5—C6—C9	109.2 (13)
O10 ^{iv} —Rb2—O11 ^{xiv}	87.3 (3)	C5—C6—O16	109.0 (13)
O10 ^{iv} —Rb2—O12 ⁱⁱⁱ	84.3 (3)	C7—C6—C9	114.3 (13)
O10 ^{iv} —Rb2—O14 ⁱⁱⁱ	87.6 (3)	C7—C6—O16	103.0 (11)
O10 ^{iv} —Rb2—O22 ^{xv}	106.7 (3)	C9—C6—O16	113.3 (12)
O11 ^v —Rb2—O11 ^{xiv}	98.2 (3)	C6—C7—C8	115.2 (13)
O11 ^v —Rb2—O12 ⁱⁱⁱ	90.4 (4)	C7—C8—O12	117.7 (15)
O11 ^v —Rb2—O14 ⁱⁱⁱ	92.4 (3)	C7—C8—O13	111.0 (14)
O11 ^v —Rb2—O22 ^{xv}	75.9 (3)	O12—C8—O13	131.3 (17)
O11 ^{xiv} —Rb2—O12 ⁱⁱⁱ	152.2 (3)	C6—C9—O14	114.9 (12)
O11 ^{xiv} —Rb2—O14 ⁱⁱⁱ	77.6 (3)	C6—C9—O15	116.7 (12)
O11 ^{xiv} —Rb2—O22 ^{xv}	76.7 (3)	O14—C9—O15	127.7 (14)
O12 ⁱⁱⁱ —Rb2—O14 ⁱⁱⁱ	75.6 (3)		

Symmetry codes: (i) $x-1, y-1, z$; (ii) $-x+1, -y+1, -z$; (iii) $x-1, y, z$; (iv) $-x+3/2, y-1/2, -z+1/2$; (v) $-x+1/2, y-1/2, -z+1/2$; (vi) $x-1/2, -y+3/2, z+1/2$; (vii) $x-1/2, -y+1/2, z+1/2$; (viii) $x+1, y+1, z$; (ix) $-x+3/2, y+1/2, -z+1/2$; (x) $-x+1/2, y+1/2, -z+1/2$; (xi) $x+1/2, -y+3/2, z-1/2$; (xii) $x+1, y, z$; (xiii) $x+1/2, -y+1/2, z-1/2$; (xiv) $x+1/2, -y+5/2, z+3/2$; (xv) $x+1/2, -y+3/2, z+3/2$.

(RAMM010_phase_2) Trirubidium citrate monohydrate

Crystal data

$3\text{Rb}^+\cdot\text{C}_6\text{H}_5\text{O}_7^{3-}\cdot\text{H}_2\text{O}$

$M_r = 28.09$

Cubic, $Fd\bar{3}m$

Hall symbol: -F 4vw 2vw

$a = 5.43105 \text{ \AA}$

$V = 160.20 \text{ \AA}^3$

$Z = 8$

$T = 300 \text{ K}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Si1	0.125	0.125	0.125	0.01*

Geometric parameters (\AA , $^\circ$)

Si1—Si1 ⁱ	2.3517	Si1—Si1 ⁱⁱⁱ	2.3517
Si1—Si1 ⁱⁱ	2.3517	Si1—Si1 ^{iv}	2.3517
Si1 ⁱ —Si1—Si1 ⁱⁱ	109.4712	Si1 ⁱⁱ —Si1—Si1 ⁱⁱⁱ	109.4712
Si1 ⁱ —Si1—Si1 ⁱⁱⁱ	109.4712	Si1 ⁱⁱ —Si1—Si1 ^{iv}	109.4712
Si1 ⁱ —Si1—Si1 ^{iv}	109.4712	Si1 ⁱⁱⁱ —Si1—Si1 ^{iv}	109.4712

Symmetry codes: (i) $x+1/4, y+1/4, -z$; (ii) $-z, x+1/4, y+1/4$; (iii) $y+1/4, -z, x+1/4$; (iv) $-x, -y, -z$.

(ramm010_DFT)

Crystal data

$3\text{Rb}^+\cdot\text{C}_6\text{H}_5\text{O}_7^{3-}\cdot\text{H}_2\text{O}$

$M_r = 463.48$

Monoclinic, $P2_1/n$

$a = 7.4477 \text{ \AA}$

$b = 11.8755 \text{ \AA}$

$c = 13.4168 \text{ \AA}$

$\beta = 97.8820^\circ$

$V = 1175.44 \text{ \AA}^3$

$Z = 4$

Cu $K\alpha$ radiation, $\lambda = 1.5418 \text{ \AA}$

$T = 300 \text{ K}$

Data collection

Density functional calculation

$h = \rightarrow$

$k = \rightarrow$

$l = \rightarrow$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Rb1	0.00583	0.17144	0.11496	0.04560*
Rb2	0.11938	0.43645	0.38752	0.04560*
Rb3	0.33332	0.44502	0.10955	0.04560*
C4	0.88224	0.90099	0.13558	0.03270*
C5	0.79490	0.79062	0.16279	0.03270*
C6	0.90539	0.68265	0.15421	0.03270*
C7	0.78654	0.58443	0.18427	0.03270*
C8	0.83169	0.47105	0.13960	0.03270*
C9	1.09412	0.68118	0.22083	0.03270*
O10	1.03887	0.92354	0.18002	0.03510*
O11	0.78874	0.96367	0.07211	0.03510*
O12	0.97854	0.42387	0.17390	0.03510*
O13	0.71389	0.43264	0.07008	0.03510*
O14	1.10018	0.68748	0.31550	0.03510*
O15	1.22967	0.66839	0.17504	0.03510*
O16	0.93669	0.67035	0.05167	0.03510*
H17	0.66841	0.77938	0.11192	0.04250*
H18	0.76254	0.79781	0.23982	0.04250*

H19	0.64492	0.60516	0.15820	0.04250*
H20	0.80258	0.57869	0.26635	0.04250*
H21	1.06826	0.65697	0.05902	0.04560*
O22	0.60639	0.21834	0.03896	0.03990*
H23	0.54454	0.20759	0.09843	0.05180*
H24	0.65210	0.29614	0.04430	0.05180*

Bond lengths (Å)

C4—C5	1.530	C7—H19	1.093
C4—O10	1.264	C7—H20	1.093
C4—O11	1.266	C8—O12	1.258
C5—C6	1.536	C8—O13	1.274
C5—H17	1.093	C9—O14	1.267
C5—H18	1.096	C9—O15	1.261
C6—C7	1.551	O16—H21	0.984
C6—C9	1.559	O22—H23	0.983
C6—O16	1.434	O22—H24	0.984
C7—C8	1.530		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O16—H21...O15	0.984	1.838	2.552	126.9
O22—H23...O14 ⁱ	0.983	1.704	2.672	168.7
O22—H24...O13	0.984	1.707	2.683	170.8
C5—H17...O22	1.093	2.674	3.749	167.4

Symmetry code: (i) $-x+3/2, y-1/2, -z+1/2$.