



Crystal structure of dirubidium hydrogen citrate from laboratory X-ray powder diffraction data and DFT comparison

Alagappa Rammohan^a and James A. Kaduk^{b*}

Received 4 December 2016

Accepted 19 December 2016

Edited by A. Van der Lee, Université de Montpellier II, France

Keywords: crystal structure; powder diffraction; density functional theory; citrate; rubidium.

CCDC references: 1523504; 1523503

Supporting information: this article has supporting information at journals.iucr.org/e

^aAtlantic International University, Honolulu, HI, USA, and ^bIllinois Institute of Technology, Chicago, IL, USA.

*Correspondence e-mail: kaduk@polycrystallography.com

The crystal structure of dirubidium hydrogen citrate, $2\text{Rb}^+\cdot\text{HC}_6\text{H}_5\text{O}_7^{2-}$, has been solved and refined using laboratory X-ray powder diffraction data, and optimized using density functional techniques. The un-ionized carboxylic acid group forms helical chains of very strong hydrogen bonds ($\text{O}\cdots\text{O} \sim 2.42 \text{ \AA}$) along the b axis. The hydroxy group participates in a chain of intra- and intermolecular hydrogen bonds along the c axis. These hydrogen bonds result in corrugated hydrogen-bonded layers in the bc plane. The Rb^+ cations are six-coordinate, and share edges and corners to form layers in the ab plane. The interlayer contacts are composed of the hydrophobic methylene groups.

1. Chemical context

In the course of a systematic study of the crystal structures of Group 1 (alkali metal) citrate salts to understand the conformational flexibility, ionization, coordination tendencies, and hydrogen bonding of the anion, 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 16 new compounds and 12 previously characterized structures are being reported separately (Rammohan & Kaduk, 2017). Six of the new structures, *i.e.* $\text{NaKHC}_6\text{H}_5\text{O}_7$, $\text{NaK}_2\text{C}_6\text{H}_5\text{O}_7$, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$, $\text{NaH}_2\text{C}_6\text{H}_5\text{O}_7$, $\text{Na}_2\text{HC}_6\text{H}_5\text{O}_7$, and $\text{K}_3\text{C}_6\text{H}_5\text{O}_7$, have been published recently (Rammohan & Kaduk, 2016*a,b,c,d,e*; Rammohan *et al.*, 2016), and two additional structures, *i.e.* $\text{KH}_2\text{C}_6\text{H}_5\text{O}_7$ and $\text{KH}_2\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})_2$, have been communicated (Kaduk & Stern, 2016*a,b*) to the Cambridge Structural Database (Groom *et al.*, 2016).

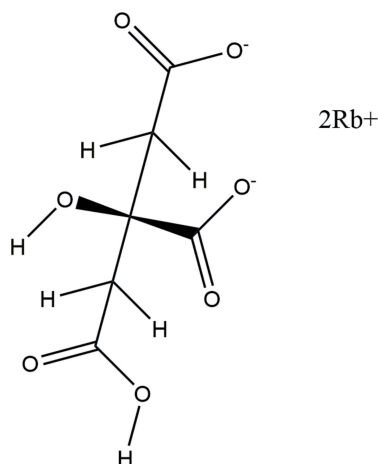
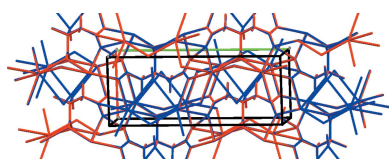


Table 1

Hydrogen-bond geometry (Å, °) for the DFT-optimized structure of dirubidium hydrogen citrate.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O11–H21 \cdots O11	1.209	1.209	2.418	180.0
O17–H18 \cdots O15	0.979	1.992	2.611	119.0
O17–H18 \cdots O16	0.979	1.992	3.216	148.6

2. Structural commentary

The asymmetric unit of the title compound is shown in Fig. 1. The r.m.s. deviation of the non-H atoms in the Rietveld refined and DFT-optimized structures is 0.052 Å (Fig. 2), and the maximum deviation is 0.083 Å, at atom C1. 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). The C1–C2–C3 angle of 111.1° is flagged as unusual [Z-score = 2.7; average = 114.3 (11)°]. The Z-score is the result of the low standard uncertainty on the average; the absolute difference of 3.2° is well within the expected range of such angles. The citrate anion occurs in the *trans,trans*-conformation, which is one of the two low-energy conformations of an isolated citrate. The central carboxylate group and the hydroxy group lie on the mirror plane. The central carboxylate O15 atom and the terminal carboxylic acid O11 atom chelate to Rb19, and the central carboxylate O16 atom and the terminal carboxylic acid O11 atom chelate to another Rb19. The Mulliken overlap populations and atomic charges indicate that the metal–oxygen bonding is ionic.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) morphology suggests that we might expect a platy morphology for dirubidium hydrogen citrate, with {020} as the principal faces. A 4th order spherical harmonic texture model was included in the refine-

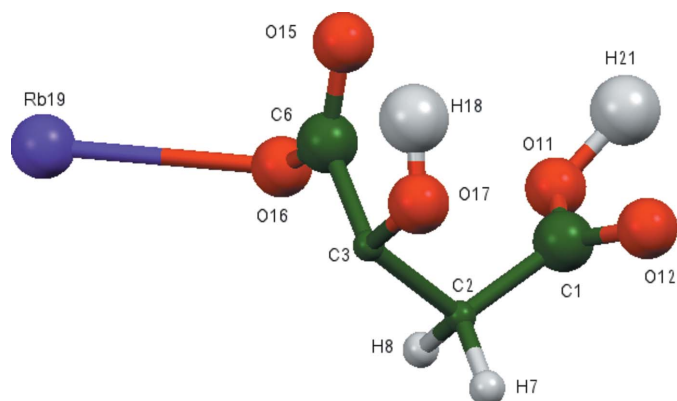


Figure 1

The asymmetric unit of the title compound, showing the atom numbering. The atoms are represented by 50% probability spheroids.

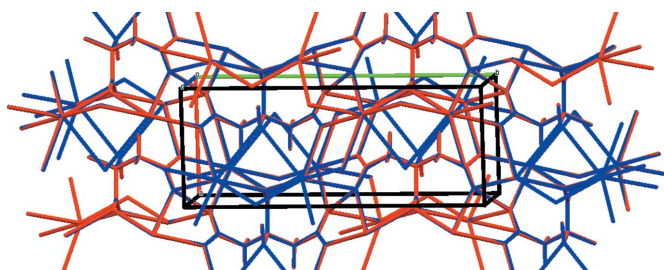


Figure 2

Comparison of the refined and optimized structures of dirubidium hydrogen citrate. The refined structure is in red and the DFT-optimized structure is in blue.

ment. The texture index was 1.078, indicating that preferred orientation was significant for this rotated flat plate specimen.

3. Supramolecular features

The Rb cation is six-coordinate (bond-valence sum = 0.96). The coordination polyhedra share corners and edges to form layers in the *ab* plane (Fig. 3). The un-ionized terminal carboxylic acid forms a very strong symmetric hydrogen bond (Table 1). The Mulliken overlap population in the hydrogen-acceptor bond is 0.161 e. By the correlation in Rammohan & Kaduk (2017), this hydrogen bond accounts for 21.9 kcal mol⁻¹ of crystal energy. The hydroxy group participates in two hydrogen bonds to ionized central carboxylate groups; one is intramolecular with graph-set motif *S*(5), and the other is intermolecular. These hydrogen bonds contribute 9.3 and 8.6 kcal mol⁻¹ to the crystal energy.

4. Database survey

Details of the comprehensive literature search for citrate structures are presented in Rammohan & Kaduk (2017). A reduced cell search of the cell of dirubidium hydrogen citrate in the Cambridge Structural Database (Groom *et al.*, 2016) (increasing the default tolerance from 1.5 to 2.0%) yielded 12 hits, but limiting the chemistry to C, H, Rb, and O only resulted in no hits. The powder pattern is now contained in the Powder Diffraction File (ICDD, 2016) as entry 00-063-1541.

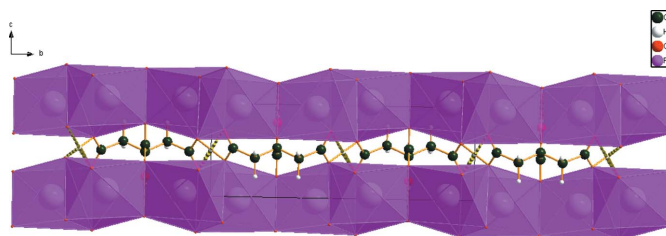


Figure 3

The crystal structure of dirubidium hydrogen citrate, viewed down the *a* axis.

Table 2
Experimental details.

	Rietveld refinement	DFT optimization
Crystal data		
Chemical formula	2Rb ⁺ ·HC ₆ H ₅ O ₇ ²⁻	2Rb ⁺ ·HC ₆ H ₅ O ₇ ²⁻
<i>M_r</i>	361.04	361.04
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>m</i>	Monoclinic, <i>P</i> 2 ₁ / <i>m</i>
Temperature (K)	300	300
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.97796 (17), 15.0960 (4), 5.32067 (19)	5.9780, 15.0961, 5.3207
β (°)	93.9341 (13)	93.9354
<i>V</i> (Å ³)	479.02 (4)	478.99
<i>Z</i>	2	2
Radiation type	<i>K</i> α ₁ , <i>K</i> α ₂ , λ = 1.540629, 1.544451 Å	–
Specimen shape, size (mm)	Flat sheet, 24 × 24	–
Data collection		
Diffractometer	Bruker D2 Phaser	–
Specimen mounting	Normal sample holder	–
Data collection mode	Reflection	–
Data collection method	Step	–
θ values (°)	$2\theta_{\min} = 5.042$ $2\theta_{\max} = 70.050$ $2\theta_{\text{step}} = 0.020$	–
Refinement		
<i>R</i> factors and goodness of fit	$R_p = 0.021$, $R_{\text{wp}} = 0.028$, $R_{\text{exp}} = 0.015$, $R(F^2) = 0.0520$, $\chi^2 = 3.312$	–
No. of parameters	49	–
No. of restraints	15	–
H-atom treatment	Only H-atom displacement parameters refined	–

Computer programs: *DIFFRAC.Measurement* (Bruker, 2009), *Endeavour* (Putz *et al.*, 1999), *GSAS* (Larson & Von Dreele, 2004), *DIAMOND* (Crystal Impact, 2015) and *pubCIF* (Westrip, 2010).

5. Synthesis and crystallization

H₃C₆H₅O₇(H₂O) (2.0768 g, 10.0 mmol) was dissolved in 10 ml deionized water. Rb₂CO₃ (10.0 mmol, 2.3170 g, Sigma–Aldrich) was added to the citric acid solution slowly with stirring. The resulting clear colorless solution was evaporated to dryness in an oven at 333 K.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Entering 22 peaks (after manually applying a constant 2θ shift to approximate specimen displacement effects) into *ITO/CRYSFIRE* (Visser, 1969; Shirley, 2002) yielded a primitive monoclinic cell having $a = 5.978$, $b = 15.096$, $c = 5.320$ Å, $\beta = 93.93^\circ$, $V = 478.33$ Å³, and $Z = 2$. Processing the pattern in *DASH3.2* (David *et al.*, 2006) suggested that the most probable space group was *P*2₁, but no acceptable solution was found. A peak list was created from the results of a Le Bail fit using the REFLIST option in *GSAS*, and imported into *Endeavour1.7b* (Putz *et al.*, 1999). Using a citrate, two Rb atoms, and the O atom of a water molecule as fragments yielded a successful structure solution. In the initial refinements, the water molecule moved very close to one of the Rb atoms, and so was removed from the refinement.

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 (Fig. 4) 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 H atoms were included at fixed positions, which were recalculated during the course of the refinement using *Materials Studio* (Dassault

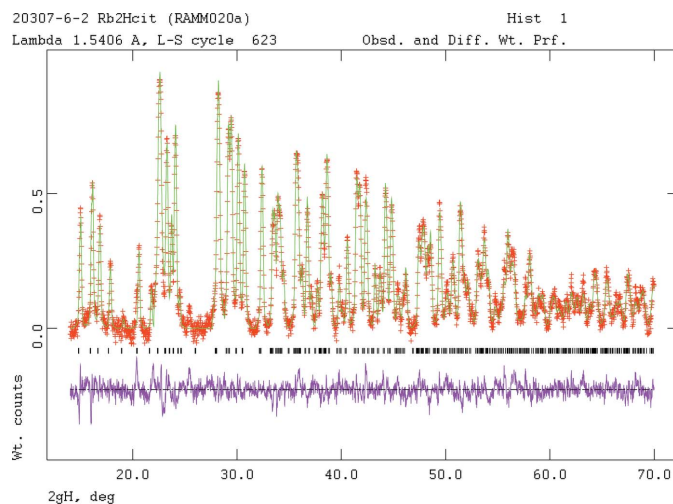


Figure 4
Rietveld plot for the refinement of dirubidium hydrogen citrate. The vertical scale is not the raw counts but the counts multiplied by the least-squares weights. This plot emphasizes the fit of the weaker peaks. The red crosses represent the observed data points and the green line is the calculated 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.

Systemes, 2014). The U_{iso} of the atoms in the central and outer portions of the citrate were constrained to be equal, and the U_{iso} of the H atoms were constrained to be 1.3 times those of the atoms to which they are attached.

The structure was solved and initially refined in the space group $P2_1$. The ADDSYM module of *PLATON* (Spek, 2009) suggested the presence of an additional centre of symmetry, and that the space group was $P2_1/m$. Refinement in this space group yielded slightly better residuals ($R_{\text{wp}} = 0.0277$ and reduced $\chi^2 = 3.3236$, compared to $R_{\text{wp}} = 0.0282$ and $\chi^2 = 3.454$ for $P2_1$), and we believe that $P2_1/m$ is the correct space group.

Stoichiometry requires one carboxylic acid proton per citrate. The space group $P2_1$ is consistent with ordered asymmetric hydrogen bonds, while $P2_1/m$ is consistent with both disordered asymmetric hydrogen bonds or symmetric hydrogen bonds. Crystallographically, it would be difficult to distinguish these two possibilities, especially using X-ray powder diffraction data. DFT calculations on the asymmetric ($P2_1$) and symmetric ($P2_1/m$) hydrogen-bond models indicate that the symmetric model is 0.2 kcal mol⁻¹ lower in energy. This difference is within the expected error range of such calculations. Since the crystallography strongly indicates the higher symmetry, we believe that the $P2_1/m$ model with symmetric hydrogen bonds is the best model for this structure.

7. DFT calculations

After the Rietveld refinement, a density functional geometry optimization (fixed experimental unit cell) was carried out using *CRYSTAL14* (Dovesi *et al.*, 2014). The basis sets for the C, H, and O atoms were those of Peintinger *et al.* (2012), and the basis set for Rb was that of Schoenes *et al.* (2008). The calculation was run on eight 2.1 GHz Xeon cores (each with 6 Gb RAM) of a 304-core Dell Linux cluster at IIT, used 8 k -points and the B3LYP functional, and took about 5 h. The U_{iso} from the Rietveld refinement were assigned to the optimized fractional coordinates.

Acknowledgements

We thank Andrey Rogachev for the use of computing resources at IIT.

References

Bravais, A. (1866). In *Etudes Cristallographiques*. Paris: Gauthier Villars.
 Bruker (2009). *DIFFRAC.Measurement*. Bruker AXS Inc., Madison, Wisconsin, USA.

Crystal Impact (2015). *DIAMOND*. Crystal Impact GbR, Bonn, Germany. <http://www.crystalimpact.com/diamond>.
 Dassault Systemes (2014). *Materials Studio*. BIOVIA, San Diego, California, USA.
 David, W. I. F., Shankland, K., van de Streek, J., Pidcock, E., Motherwell, W. D. S. & Cole, J. C. (2006). *J. Appl. Cryst.* **39**, 910–915.
 Donnay, J. D. H. & Harker, D. (1937). *Am. Mineral.* **22**, 446–467.
 Dovesi, R., Orlando, R., Erba, A., Zicovich-Wilson, C. M., Civalleri, B., Casassa, S., Maschio, L., Ferrabone, M., De La Pierre, M., D'Arco, P., Noel, Y., Causa, M., Rerat, M. & Kirtman, B. (2014). *Int. J. Quantum Chem.* **114**, 1287–1317.
 Finger, L. W., Cox, D. E. & Jephcoat, A. P. (1994). *J. Appl. Cryst.* **27**, 892–900.
 Friedel, G. (1907). *Bull. Soc. Fr. Mineral.* **30**, 326–455.
 Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
 Howard, C. J. (1982). *J. Appl. Cryst.* **15**, 615–620.
 ICDD (2016). *PDF-4+ 2015 and PDF-4 Organics 2016 (Databases)*, edited by S. Kabekkodu. International Centre for Diffraction Data, Newtown Square PA, USA.
 Kaduk, J. A. & Stern, C. (2016a). CSD Communication 1446457-1446458. CCDC, Cambridge, England.
 Kaduk, J. A. & Stern, C. (2016b). CSD Communication 1446460-1446461. CCDC, Cambridge, England.
 Larson, A. C. & Von Dreele, R. B. (2004). *General Structure Analysis System (GSAS)*. Report LAUR 86-784 Los Alamos National Laboratory, New Mexico, USA.
 Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
 Peintinger, M. F., Vilela Oliveira, D. & Bredow, T. (2012). *J. Comput. Chem.* **34**, 451–459.
 Putz, H., Schön, J. C. & Jansen, M. (1999). *J. Appl. Cryst.* **32**, 864–870.
 Rammohan, A. & Kaduk, J. A. (2017). Submitted to *Acta Cryst.* **B72** [hw5042].
 Rammohan, A. & Kaduk, J. A. (2016a). *Acta Cryst.* **E72**, 170–173.
 Rammohan, A. & Kaduk, J. A. (2016b). *Acta Cryst.* **E72**, 403–406.
 Rammohan, A. & Kaduk, J. A. (2016c). *Acta Cryst.* **E72**, 793–796.
 Rammohan, A. & Kaduk, J. A. (2016d). *Acta Cryst.* **E72**, 854–857.
 Rammohan, A. & Kaduk, J. A. (2016e). *Acta Cryst.* **E72**, 1159–1162.
 Rammohan, A., Sarjeant, A. A. & Kaduk, J. A. (2016). *Acta Cryst.* **E72**, 943–946.
 Schoenes, J., Racu, A.-M., Doll, K., Bukowski, Z. & Karpinski, J. (2008). *Phys. Rev. B*, **77**, 134515.
 Shirley, R. (2002). *The Crysfire 2002 system for automatic powder indexing*. Guildford, Surrey, England.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Stephens, P. W. (1999). *J. Appl. Cryst.* **32**, 281–289.
 Streek, J. van de & Neumann, M. A. (2014). *Acta Cryst.* **B70**, 1020–1032.
 Thompson, P., Cox, D. E. & Hastings, J. B. (1987). *J. Appl. Cryst.* **20**, 79–83.
 Toby, B. H. (2001). *J. Appl. Cryst.* **34**, 210–213.
 Visser, J. W. (1969). *J. Appl. Cryst.* **2**, 89–95.
 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2017). E73, 92-95 [https://doi.org/10.1107/S2056989016020168]

Crystal structure of dirubidium hydrogen citrate from laboratory X-ray powder diffraction data and DFT comparison

Alagappa Rammohan and James A. Kaduk

Computing details

Data collection: *DIFFRAC.Measurement* (Bruker, 2009) for RAMM020C_publ. Program(s) used to solve structure: *Endeavour* (Putz *et al.*, 1999) for RAMM020C_publ. Program(s) used to refine structure: *GSAS* for RAMM020C_publ. Molecular graphics: *DIAMOND* (Crystal Impact, 2015) for RAMM020C_publ. Software used to prepare material for publication: *pubCIF* (Westrip, 2010) for RAMM020C_publ.

(RAMM020C_publ) Dirubidium hydrogen citrate

Crystal data

$2\text{Rb}^+\cdot\text{HC}_6\text{H}_5\text{O}_7^{2-}$

$M_r = 361.04$

Monoclinic, $P2_1/m$

Hall symbol: -P 2yb

$a = 5.97796$ (17) Å

$b = 15.0960$ (4) Å

$c = 5.32067$ (19) Å

$\beta = 93.9341$ (13)°

$V = 479.02$ (4) Å³

$Z = 2$

$D_x = 2.503$ Mg m⁻³

$K\alpha_1, K\alpha_2$ radiation, $\lambda = 1.540629, 1.544451$ Å

$T = 300$ K

white

flat_sheet, 24 × 24 mm

Specimen preparation: Prepared at 333 K

Data collection

Bruker D2 Phaser

diffractometer

Specimen mounting: Normal sample holder

Data collection mode: reflection

Scan method: step

$2\theta_{\min} = 5.042^\circ$, $2\theta_{\max} = 70.050^\circ$, $2\theta_{\text{step}} = 0.020^\circ$

Refinement

Least-squares matrix: full

$R_p = 0.021$

$R_{\text{wp}} = 0.028$

$R_{\text{exp}} = 0.015$

$R(F^2) = 0.0520$

3217 data points

Profile function: CW Profile function number 4
with 21 terms Pseudovoigt profile coefficients
as parameterized in P. Thompson, D.E. Cox &
J.B. Hastings (1987). J. Appl. Cryst.,20,79-83.
Asymmetry correction of L.W. Finger, D.E. Cox
& A. P. Jephcoat (1994). J. Appl.
Cryst.,27,892-900. Microstrain broadening by
P.W. Stephens, (1999). J. Appl.
Cryst.,32,281-289. #1(GU) = 142.783 #2(GV) =
0.000 #3(GW) = 4.751 #4(GP) = 0.000 #5(LX)
= 5.874 #6(ptec) = -0.71 #7(trns) = 1.83 #8(shft)
= -25.7226 #9(sfec) = 0.00 #10(S/L) = 0.0441
#11(H/L) = 0.0005 #12(eta) = 0.0000 #13(S400
) = 3.9E-02 #14(S040) = 8.9E-05 #15(S004) =
3.1E-01 #16(S220) = 9.7E-03 #17(S202) =
-6.4E-02 #18(S022) = 5.7E-04 #19(S301) =
-8.2E-03 #20(S103) = 2.5E-02 #21(S121) =
-8.8E-03 Peak tails are ignored where the
intensity is below 0.0100 times the peak Aniso.
broadening axis 0.0 1.0 0.0
49 parameters
15 restraints
Only H-atom displacement parameters refined
Weighting scheme based on measured s.u.'s
 $(\Delta/\sigma)_{\max} = 0.08$
Background function: GSAS Background
function number 1 with 4 terms. Shifted
Chebyshev function of 1st kind 1: 3185.87 2:
-310.317 3: -87.5319 4: 63.9418

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2727 (12)	0.4151 (4)	0.5145 (14)	0.0556 (15)*
C2	0.3859 (11)	0.33308 (16)	0.4236 (14)	0.026 (4)*
C3	0.2821 (12)	0.25	0.5368 (15)	0.026 (4)*
C6	0.0268 (13)	0.25	0.463 (2)	0.0556 (15)*
H7	0.56395	0.33136	0.47541	0.018 (5)*
H8	0.35795	0.33169	0.21841	0.018 (5)*
O11	0.1036 (11)	0.4467 (5)	0.3841 (15)	0.0556 (15)*
O12	0.3502 (12)	0.4512 (5)	0.7178 (14)	0.0556 (15)*
O15	-0.0968 (16)	0.25	0.650 (2)	0.0556 (15)*
O16	-0.0415 (16)	0.25	0.232 (2)	0.0556 (15)*
O17	0.3416 (14)	0.25	0.7999 (15)	0.0556 (15)*
H18	0.17127	0.25	0.87091	0.068 (2)*
Rb19	-0.2264 (3)	0.10523 (10)	-0.0313 (3)	0.0590 (9)*
H21	0.0	0.0	0.5	0.07*

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

C1—C2	1.5070 (17)	O12—Rb19 ^{iv}	2.907 (7)
C1—O11	1.278 (5)	O15—C6	1.281 (5)
C1—O12	1.270 (5)	O15—Rb19 ^v	2.903 (7)

C2—C1	1.5070 (17)	O15—Rb19 ^{vi}	2.903 (7)
C2—C3	1.5400 (17)	O16—C6	1.267 (5)
C2—H7	1.081 (7)	O16—Rb19	2.784 (7)
C2—H8	1.093 (7)	O16—Rb19 ⁱ	2.784 (7)
C3—C2	1.5400 (17)	O17—C3	1.420 (5)
C3—C2 ⁱ	1.5400 (17)	O17—H18	1.110 (8)
C3—C6	1.5499 (18)	O17—Rb19 ^{vii}	3.454 (7)
C3—O17	1.420 (5)	O17—Rb19 ^{iv}	3.455 (7)
C6—C3	1.5499 (18)	H18—O17	1.110 (8)
C6—O15	1.281 (5)	Rb19—O11 ^{viii}	3.159 (7)
C6—O16	1.267 (5)	Rb19—O11 ⁱ	2.965 (7)
H7—C2	1.081 (7)	Rb19—O12 ^{ix}	2.984 (6)
H8—C2	1.093 (8)	Rb19—O12 ^x	2.907 (7)
O11—C1	1.278 (5)	Rb19—O15 ^{xi}	2.903 (7)
O11—Rb19 ⁱⁱ	3.159 (7)	Rb19—O16	2.784 (7)
O11—Rb19 ⁱ	2.965 (7)	Rb19—O17 ^{xii}	3.454 (7)
O11—H21 ⁱⁱⁱ	1.209 (6)	H21—O11 ^{ix}	1.209 (6)
O12—C1	1.270 (5)	H21—O11 ⁱ	1.209 (6)
O12—Rb19 ⁱⁱⁱ	2.984 (6)		
C2—C1—O11	119.2 (4)	C6—O15—Rb19 ^v	130.21 (19)
C2—C1—O12	118.7 (4)	C6—O15—Rb19 ^{xv}	130.21 (19)
O11—C1—O12	122.1 (4)	Rb19 ^v —O15—Rb19 ^{xv}	97.7 (3)
C1—C2—C3	110.0 (4)	C6—O16—Rb19	125.3 (2)
C1—C2—H7	113.2 (4)	C6—O16—Rb19 ^{xiii}	125.3 (2)
C1—C2—H8	17.29 (4)	Rb19—O16—Rb19 ^{xiii}	103.5 (4)
C3—C2—H7	107.3 (5)	C3—O17—H18	99.3 (7)
C3—C2—H8	109.7 (5)	O11 ^{viii} —Rb19—O11 ^{xiii}	94.2 (2)
H7—C2—H8	109.5 (5)	O11 ^{viii} —Rb19—O12 ^{ix}	79.54 (16)
C2—C3—C2 ^{xiii}	109.1 (4)	O11 ^{viii} —Rb19—O12 ^{xvi}	74.7 (2)
C2—C3—C6	108.5 (4)	O11 ^{viii} —Rb19—O15 ^{xi}	98.11 (18)
C2—C3—O17	107.8 (4)	O11 ^{viii} —Rb19—O16	142.5 (2)
C2 ^{xiii} —C3—C6	108.5 (4)	O11 ^{xiii} —Rb19—O12 ^{ix}	63.59 (19)
C2 ^{xiii} —C3—O17	107.8 (4)	O11 ^{xiii} —Rb19—O12 ^{xvi}	141.13 (14)
C6—C3—O17	115.2 (7)	O11 ^{xiii} —Rb19—O15 ^{xi}	116.3 (2)
C3—C6—O15	114.4 (9)	O11 ^{xiii} —Rb19—O16	67.2 (2)
C3—C6—O16	119.5 (9)	O12 ^{ix} —Rb19—O12 ^{xvi}	77.7 (2)
O15—C6—O16	126.1 (9)	O12 ^{ix} —Rb19—O15 ^{xi}	177.6 (2)
C1—O11—Rb19 ⁱⁱ	113.5 (7)	O12 ^{ix} —Rb19—O16	115.8 (3)
C1—O11—Rb19 ^{xiii}	140.0 (4)	O12 ^{xvi} —Rb19—O15 ^{xi}	102.2 (2)
Rb19 ⁱⁱ —O11—Rb19 ^{xiii}	85.8 (2)	O12 ^{xvi} —Rb19—O16	139.9 (2)
C1—O12—Rb19 ⁱⁱⁱ	136.0 (4)	O15 ^{xi} —Rb19—O16	65.8 (2)
C1—O12—Rb19 ^{xiv}	121.4 (4)	O11 ^{ix} —H21—O11 ^{xiii}	180.0
Rb19 ⁱⁱⁱ —O12—Rb19 ^{xiv}	102.3 (2)		

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

(ramm020c_DFT)

Crystal data

$C_6H_6O_7Rb_2$	$c = 5.3207 \text{ \AA}$
$M_r = 361.04$	$\beta = 93.9354^\circ$
Monoclinic, $P2_1/m$	$V = 478.99 \text{ \AA}^3$
Hall symbol: $-P 2yb$	$Z = 2$
$a = 5.9780 \text{ \AA}$	$T = 300 \text{ K}$
$b = 15.0961 \text{ \AA}$	

Data collection

$h = \rightarrow$	$l = \rightarrow$
$k = \rightarrow$	

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

	x	y	z	U_{iso}^*/U_{eq}
C1	0.28187	0.41704	0.52143	0.05270*
C2	0.38508	0.33363	0.42243	0.01400*
C3	0.27797	0.25000	0.53116	0.01400*
C6	0.02276	0.25000	0.45797	0.05270*
H7	0.56395	0.33136	0.47541	0.01820*
H8	0.35795	0.33169	0.21841	0.01820*
O11	0.10613	0.44671	0.38734	0.05270*
O12	0.35379	0.45283	0.72015	0.05270*
O15	-0.10430	0.25000	0.63801	0.05270*
O16	-0.03680	0.25000	0.22772	0.05270*
O17	0.31806	0.25000	0.79999	0.05270*
H18	0.17127	0.25000	0.87091	0.06840*
Rb19	-0.22414	0.10319	-0.03836	0.05980*
H21	0.00000	0.00000	0.50000	0.07000*

Bond lengths (\AA)

C1—C2	1.513	Rb19—O11 ⁱⁱ	2.997
C1—O11	1.308	Rb19—O16	2.820
C1—O12	1.238	Rb19—O12 ^{viii}	2.966
C2—C3	1.546	C3—C2 ⁱⁱ	1.546
C2—H7	1.087	C3—C6	1.548
C2—H8	1.087	C3—O17	1.434
O11—Rb19 ⁱ	3.115	C6—O15	1.263
O11—Rb19 ⁱⁱ	2.997	C6—O16	1.252
O11—H21 ⁱⁱⁱ	1.209	O15—Rb19 ^{ix}	2.926
O12—Rb19 ^{iv}	2.879	O15—Rb19 ^x	2.926
O12—Rb19 ⁱⁱⁱ	2.966	O16—Rb19 ⁱⁱ	2.820
Rb19—O12 ^v	2.879	O17—H18	0.979
Rb19—O15 ^{vi}	2.926	H21—O11 ⁱⁱ	1.209
Rb19—O11 ^{vii}	3.115	H21—O11 ^{viii}	1.209

Symmetry codes: (i) $-x, y+1/2, -z$; (ii) $x, -y+1/2, z$; (iii) $-x, y+1/2, -z+1$; (iv) $x+1, -y+1/2, z+1$; (v) $x-1, -y+1/2, z-1$; (vi) $x, y, z-1$; (vii) $-x, y-1/2, -z$; (viii) $-x, y-1/2, -z+1$; (ix) $x, y, z+1$; (x) $x, -y+1/2, z+1$.

Hydrogen-bond geometry (Å, °)

<i>D—H···A</i>	<i>D—H</i>	<i>H···A</i>	<i>D···A</i>	<i>D—H···A</i>
O11—H21···O11	1.209	1.209	2.418	180.0
O17—H18···O15	0.979	1.992	2.611	119.0
O17—H18···O16	0.979	1.992	3.216	148.6