



Crystal structures of two isostructural compounds: a second polymorph of dipotassium hydrogen citrate, $K_2HC_6H_5O_7$, and potassium rubidium hydrogen citrate, $KRbHC_6H_5O_7$

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The crystal structures of a new polymorph of dipotassium hydrogen citrate, $2K^+ \cdot HC_6H_5O_7^{2-}$, and potassium rubidium hydrogen citrate, $K^+ \cdot Rb^+ \cdot HC_6H_5O_7^{2-}$, have been solved and refined using laboratory powder X-ray diffraction and optimized using density functional techniques. In the new polymorph of the dipotassium salt, KO_7 and KO_8 coordination polyhedra share corners and edges to form a three-dimensional framework with channels parallel to the a axis and $[111]$. The hydrophobic methylene groups face each other in the channels. The un-ionized carboxylic acid group forms a strong charge-assisted hydrogen bond to the central ionized carboxylate group. The hydroxy group forms an intermolecular hydrogen bond to a different central carboxylate group. In the potassium rubidium salt, the K^+ and Rb^+ cations are disordered over two sites, in approximately 0.72:0.28 and 0.28:0.72 ratios. KO_8 and RbO_9 coordination polyhedra share corners and edges to form a three-dimensional framework with channels parallel to the a axis. The un-ionized carboxylic acid group forms a strong charge-assisted hydrogen bond to an ionized carboxylate group. The hydroxy group forms an intermolecular hydrogen bond to the central carboxylate group. Density functional theory (DFT) calculations on the ordered cation structures suggest that interchange of K^+ and Rb^+ at the two cation sites changes the energy insignificantly.

1. Introduction

A systematic study of the crystal structures of Group 1 (alkali metal) citrate salts has been reported in Rammohan & Kaduk (2018). The study was extended to lithium metal hydrogen citrates in Cigler & Kaduk (2018), to sodium metal hydrogen citrates in Cigler & Kaduk (2019*a*), to sodium dirubidium citrates in Cigler & Kaduk (2019*b*), to dilithium potassium citrate in Cigler & Kaduk (2019*c*), to lithium dipotassium citrate monohydrate in Cigler & Kaduk (2020), to ammonium citrates in Wheatley & Kaduk (2019), to diammonium sodium citrate in Bhaskar *et al.* (2020), to diammonium potassium citrate in Patel *et al.* (2020), and to disodium hydrogen citrate monohydrate in Hong *et al.* (2020). These previous studies have established the normal conformations of a citrate anion, and illuminated the trends in its coordination to cations. They have permitted the establishment of a correlation between the Mulliken overlap population and the energy of $O-H \cdots O$ hydrogen bonds, and confirmed that the normal order of ionization of the carboxyl groups is: central, one terminal, and second terminal. Establishing the normal patterns of citrate structures permits easier interpretation of new structures in that context. The compounds reported here, namely, dipo-

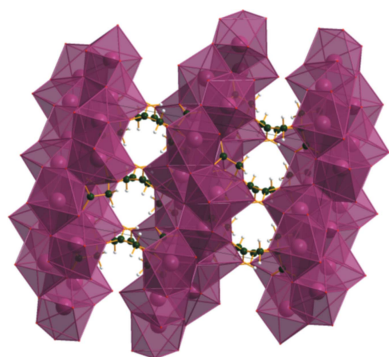


Table 1
Experimental details.

	kadu1878 (phase 0)	kadu1878 (DFT)	kadu1860 (phase 0)	ZZZQRO01 (DFT)
Crystal data				
Chemical formula	2K ⁺ ·C ₆ H ₆ O ₇ ²⁻	2K ⁺ ·C ₆ H ₆ O ₇ ²⁻	2K ⁺ ·C ₆ H ₆ O ₇ ²⁻	2K ⁺ ·C ₆ H ₆ O ₇ ²⁻
<i>M_r</i>	268.3	268.3	268.3	268.3
Crystal system, space group	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>C2/c</i>	Monoclinic, <i>C2/c</i>
Temperature (K)	304	–	300	–
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.3473 (3), 16.1428 (10), 11.0103 (8)	5.3473, 16.1429, 11.0103	10.7735 (6), 9.6647 (5), 18.9330 (8)	10.7570, 9.6500, 18.9180
β (°)	91.438 (4)	91.4375	90.045 (6)	90.0900
<i>V</i> (Å ³)	950.12 (13)	950.12	1971.4 (2)	1963.78
<i>Z</i>	4	4	8	8
Specimen shape, size (mm)	Cylinder, 12 × 0.7	–	Flat sheet, 25 × 25	–
Data collection				
Diffractometer	PANalytical Empyrean	DFT calculation	Bruker D2 Phaser	DFT calculation
Specimen mounting	Glass capillary	–	PMMA holder with Kapton film	–
Data collection mode	Transmission	–	Reflection	–
Data collection method	Step	–	Step	–
Refinement				
No. of restraints	58	–	0	–
	acig037	acig037_19 (DFT)	acig037_20 (DFT)	kadu1880 (phase 0)
Crystal data				
Chemical formula	0.989K ⁺ ·1.011Rb ⁺ ·C ₆ H ₆ O ₇ ²⁻	K ⁺ ·Rb ⁺ ·C ₆ H ₆ O ₇ ²⁻	K ⁺ ·Rb ⁺ ·C ₆ H ₆ O ₇ ²⁻	1.668K ⁺ ·0.332Rb ⁺ ·C ₆ H ₆ O ₇ ²⁻
<i>M_r</i>	315.16	314.6	314.6	283.68
Crystal system, space group	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>C2/c</i>
Temperature (K)	300	–	–	300
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.3500 (6), 16.2800 (13), 11.1735 (15)	5.3536, 16.2869, 11.1813	5.3536, 16.2869, 11.1813	10.8409 (7), 9.6926 (6), 18.9810 (13)
β (°)	91.058 (7)	91.0480	91.0480	90.150 (6)
<i>V</i> (Å ³)	973.0 (2)	974.77	974.77	1994.4 (2)
<i>Z</i>	4	4	4	8
Radiation type	K α _{1,2}	–	–	–
Specimen shape, size (mm)	–	–	–	Flat sheet, 25 × 25
Data collection				
Diffractometer	–	DFT calculation	DFT calculation	Bruker D2 Phaser
Specimen mounting	–	–	–	PMMA holder
Data collection mode	–	–	–	Reflection
Data collection method	–	–	–	Step
θ values (°)	2 θ _{min} = 1.021 2 θ _{max} = 49.985 2 θ _{step} = 0.017	–	–	–
Refinement				
<i>R</i> factors and goodness of fit	<i>R</i> _p = 0.038, <i>R</i> _{wp} = 0.049, <i>R</i> _{exp} = 0.018, <i>R</i> (<i>F</i> ²) = 0.08896, χ^2 = 7.779	–	–	–
No. of parameters	72	–	–	–
No. of restraints	–	–	–	0

Computer programs: *GSAS-II* (Toby & Von Dreele, 2013).

tassium hydrogen citrate, K₂HC₆H₅O₇, and potassium rubidium hydrogen citrate, KRbHC₆H₅O₇, represent further extensions to the series (Scheme 1).

2. Experimental

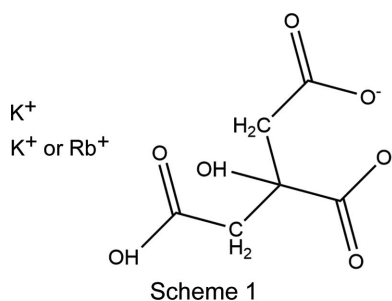
2.1. Synthesis and crystallization

2.1.1. Dipotassium hydrogen citrate, K₂HC₆H₅O₇. Dipotassium hydrogen citrate was synthesized inadvertently in an attempt to prepare dipotassium ammonium citrate. Citric acid monohydrate (1.0351 g; Sigma–Aldrich, Lot No. 089K0091), potassium carbonate (0.6772 g; Sigma–Aldrich, Lot No. 098K0064), and ammonium carbonate (0.2390 g; Aldrich)

were dissolved in water (10 ml). When the effervescence stopped, the clear solution was dried in a 368 K oven. After 9 h, the slightly damp solid was removed from the oven and dried overnight under ambient conditions. The yield was 98.7% of white solid. After one month of storage under ambient conditions, the sample changed into a known polymorph [Cambridge Structural Database (Groom *et al.*, 2016) refcode ZZZQRO01 (Zacharias & Glusker, 1993)].

2.1.2. Potassium rubidium hydrogen citrate, KRbHC₆H₅O₇. Potassium rubidium hydrogen citrate was synthesized by adding stoichiometric quantities of K₂CO₃ (Sigma–Aldrich) and Rb₂CO₃ (Sigma–Aldrich) to a solution of citric acid monohydrate (2.03 g, 10.0 mmol; Sigma–Aldrich) in water (10 ml). After the effervescence subsided, the clear solution

was dried in a 403 K oven to yield a pale-yellow solid. After storage under ambient conditions for three years, the compound changed into a mixture of phases.



2.2. Refinement

2.2.1. Dipotassium hydrogen citrate, $K_2HC_6H_5O_7$. Crystal data, data collection and structure refinement details for dipotassium hydrogen citrate (kadu1860) are summarized in Table 1. A Rietveld plot is presented in Fig. 1. Initial attempts to index the pattern using *DICVOL14* (Louër & Boulton, 2014) yielded a triclinic cell with $a = 9.8657$, $b = 10.8485$, $c = 11.0558$ Å, $\alpha = 113.149$, $\beta = 100.948$, $\gamma = 108.864^\circ$, $V = 958.80$ Å³, and $Z = 4$. Although this yielded a good Le Bail fit, attempts to solve the structure with *FOX* (Favre-Nicolin & Černý, 2002) led to poor cost factors. Repeating the preparation resulted in a pattern with many common peaks, but some peaks decreased in intensity. Indexing the common peaks in *JADE Pro* (MDI, 2019) yielded a $P2_1/c$ unit cell with $a = 5.345$, $b = 16.143$, $c = 11.008$ Å, $\beta = 91.50^\circ$, $V = 949.6$ Å³, and $Z = 4$. A Le Bail fit in *FOX* yielded $R_{wp} = 5.939\%$. Using *FOX* to solve the structure with a citrate, 2K, and N as fragments yielded a

low-cost factor, but the N was too close to the citrate and was removed from the model. The unit cell was very close to that of potassium rubidium hydrogen citrate (Cigler & Kaduk, 2020), so we concluded that the current compound is a new polymorph of $K_2HC_6H_5O_7$ (Zacharias & Glusker, 1993). The $KRbHC_6H_5O_7$ model was used to begin refinement. The variable peaks corresponded to $(NH_4)_2KC_6H_5O_7$ (Patel *et al.*, 2020).

The structure was refined by the Rietveld method using *GSAS-II* (Toby & Von Dreele, 2013). The H atoms were included in fixed positions, which were recalculated during the course of the refinement using *Materials Studio* (Dassault Systems, 2019). All C–C and C–O bond lengths, and all bond angles were restrained based on a *Mercury/Mogul Geometry Check* (Sykes *et al.*, 2011; Bruno *et al.*, 2004) of the molecule. The U_{iso} values of the atoms in the central and outer portions of the citrate anion were constrained to be equal, and the U_{iso} values of the H atoms were constrained to be 1.3 times those of the atoms to which they are attached. A Chebyshev polynomial function with four coefficients, along with a peak at 13.11° to model the scattering of the glass capillary, was used to model the background. The refinement showed that the sample was a mixture of 86.0% $K_2HC_6H_5O_7$ and 14.0% $(NH_4)_2KC_6H_5O_7$ (Patel *et al.*, 2020).

A density functional geometry optimization was carried out using *CRYSTAL14* (Dovesi *et al.*, 2014). The basis sets for the H, C, and O atoms were those of Gatti *et al.* (1994), and the basis set for K was that of Peintinger *et al.* (2013). 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, using 8 k -points and the B3LYP functional, and took ~ 4 d. A similar optimization was carried out for ZZQRO01.

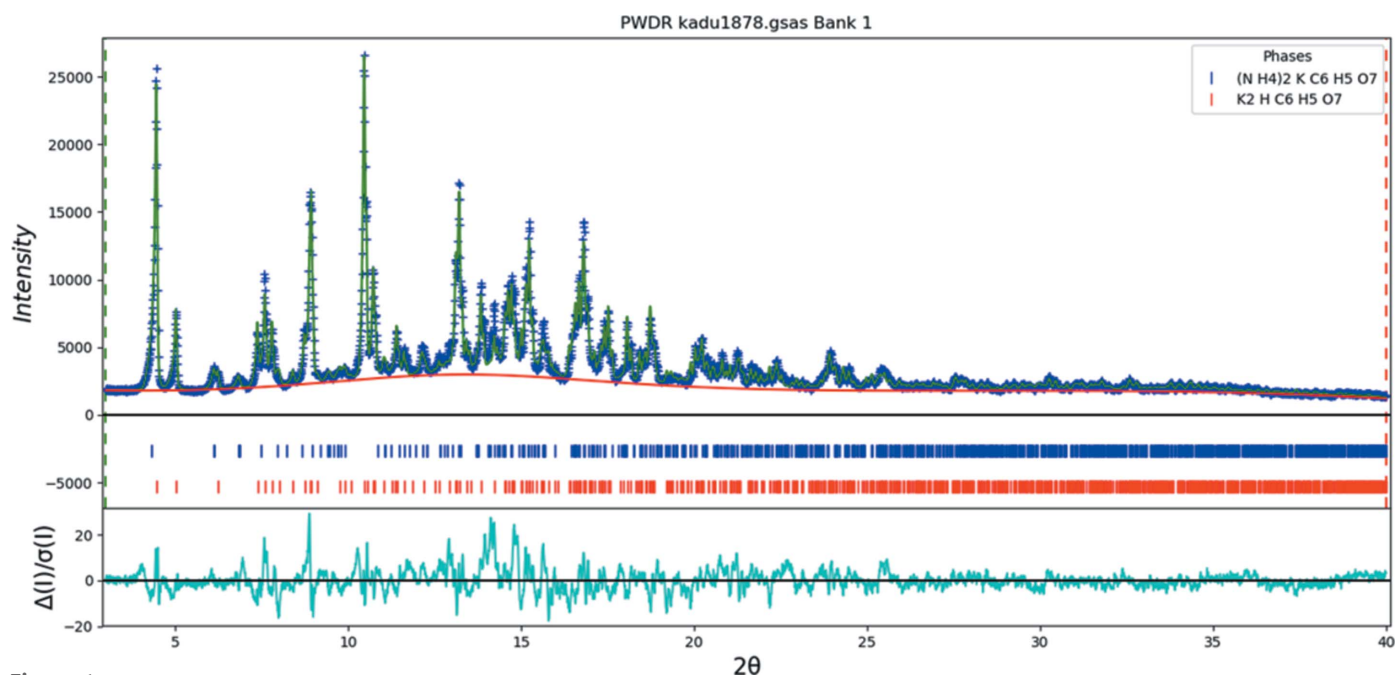


Figure 1 Rietveld plot for the new polymorph of $K_2HC_6H_5O_7$. The blue crosses represent the observed data points and the green line is the calculated pattern. The cyan curve is the normalized error plot. The row of red tick marks indicates the calculated reflection positions for the major phase, and the row of blue tick marks indicates the reflection positions for the $(NH_4)_2KC_6H_5O_7$ impurity. The red line is the background curve.

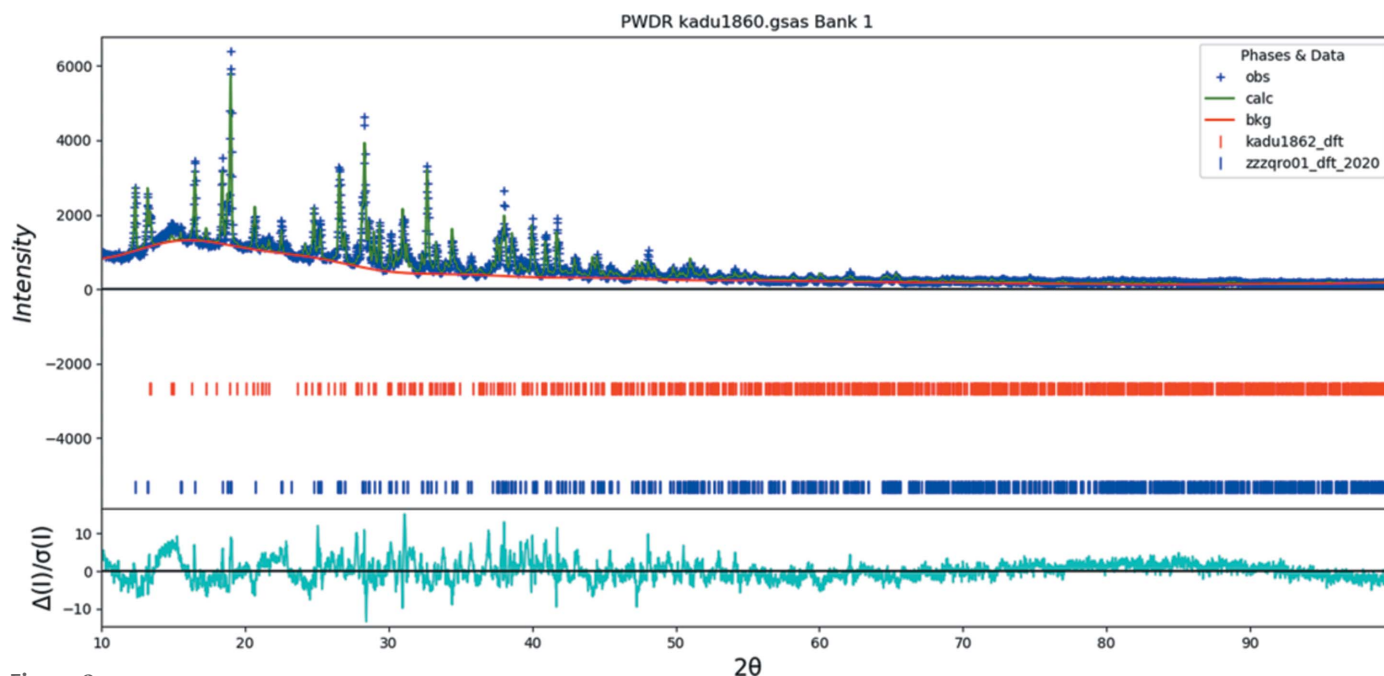


Figure 2

Rietveld plot for $\text{K}_2\text{HC}_6\text{H}_5\text{O}_7$ after storage. The blue crosses represent the observed data points and the green line is the calculated pattern. The cyan curve is the normalized error plot. The row of blue tick marks indicates the calculated reflection positions for the major phase, and the row of red tick marks indicates the reflection positions for the $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$ impurity. The red line is the background curve.

A Rietveld refinement of the aged sample (kadu1860) showed it to be a mixture of 80% ZZZQRO01 and 20% $(\text{NH}_4)_2\text{KC}_6\text{H}_5\text{O}_7$ (Patel *et al.*, 2020) (Fig. 2).

2.2.2. Potassium rubidium hydrogen citrate, $\text{KRbHC}_6\text{H}_5\text{O}_7$. Crystal data, data collection and structure refinement details for potassium rubidium hydrogen citrate (acig037) are summarized in Table 1 (Fig. 3). To minimize Rb fluorescence, the

pulse height discriminator lower level of the X'Celerator detector was raised from the default 39.0 to 51.0%. The structure was solved using Monte Carlo simulated annealing techniques with *FOX* (Favre-Nicolin & Černý, 2002) using a citrate anion, one K atom, and one Rb atom as fragments. The structure was refined by the Rietveld method using *GSAS-II* (Toby & Von Dreele, 2013). The sample was highly absorbing

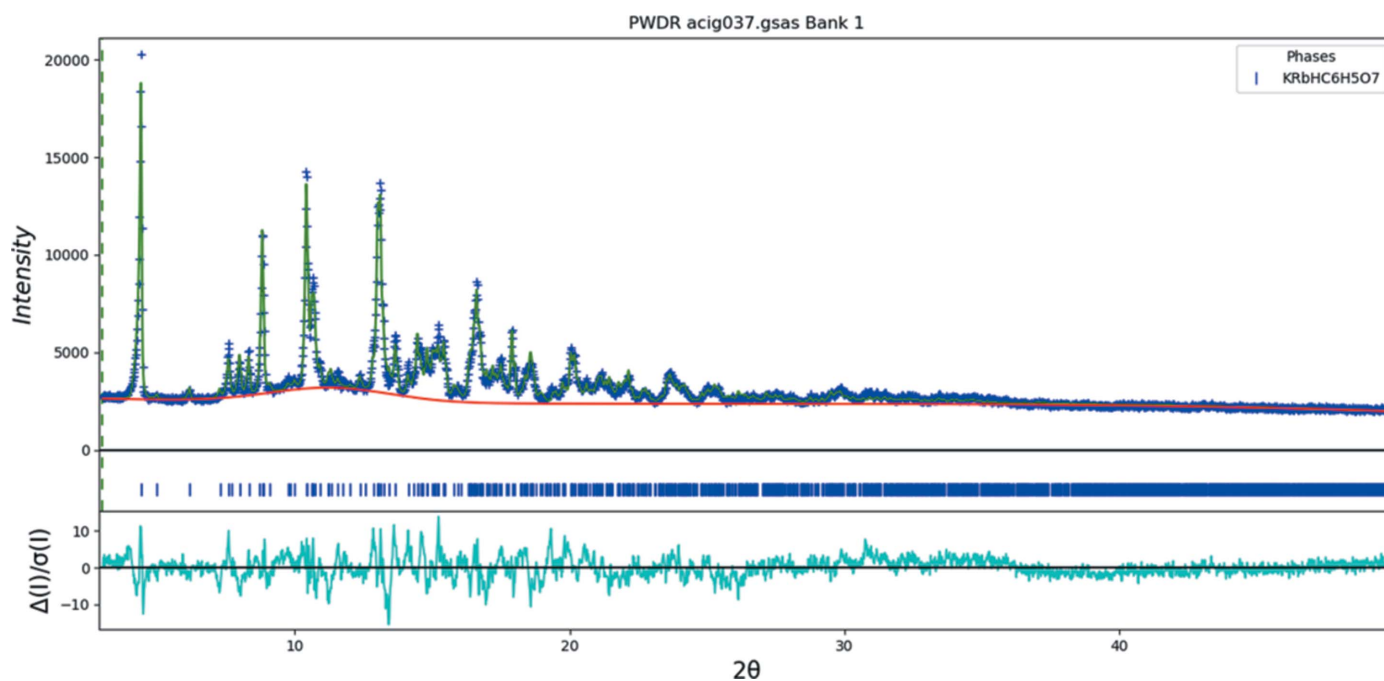


Figure 3

Rietveld plot for $\text{KRbHC}_6\text{H}_5\text{O}_7$. The blue crosses represent the observed data points and the green line is the calculated pattern. The cyan curve is the normalized error plot. The row of blue tick marks indicates the calculated reflection positions for the major phase. The red line is the background curve.

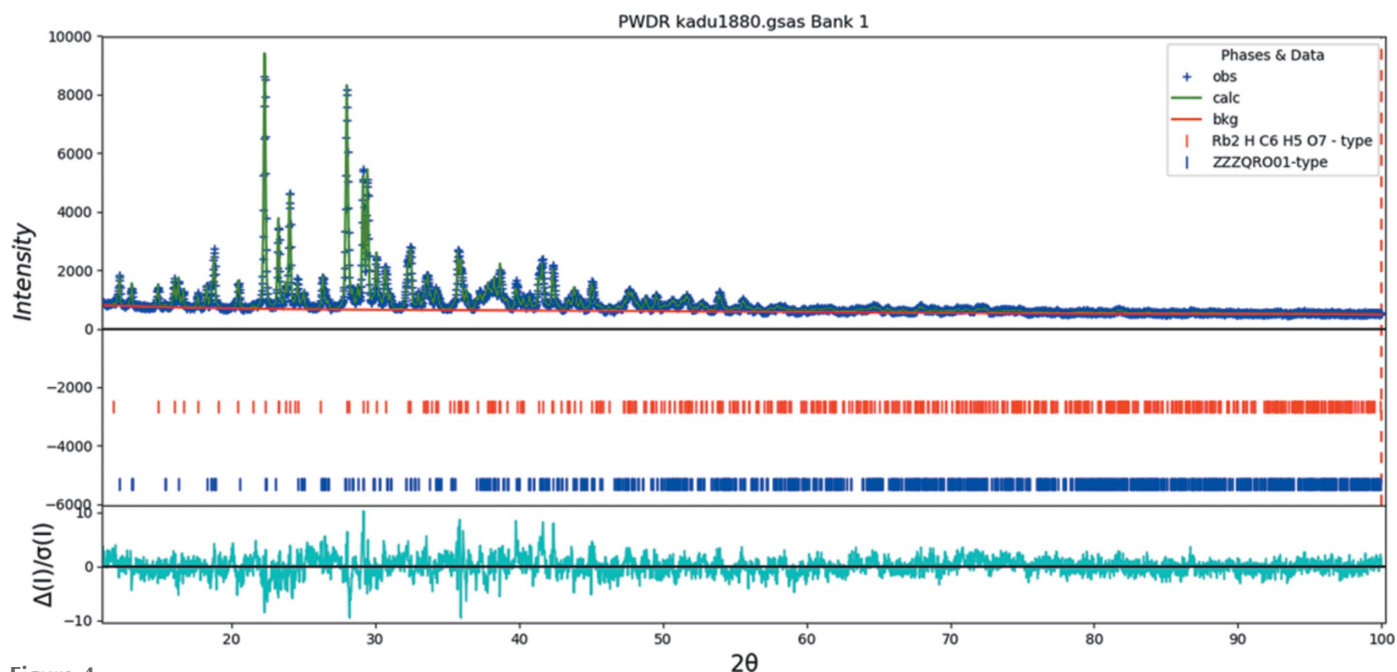


Figure 4
Rietveld plot for $\text{KRbHC}_6\text{H}_5\text{O}_7$ after storage. The blue crosses represent the observed data points and the green line is the calculated pattern. The cyan curve is the normalized error plot. The row of blue tick marks indicates the calculated reflection positions for the K-major phase and the row of red tick marks indicates the reflection positions for the Rb-major phase. The red line is the background curve.

($\mu \cdot R = 7.17$) and an absorption correction was included. The H atoms were included in fixed positions, which were recalculated during the course of the refinement using *Materials Studio* (Dassault Systems, 2019). All C–C and C–O bond lengths, and all bond angles were restrained based on a *Mercury/Mogul Geometry Check* (Sykes *et al.*, 2011; Bruno *et al.*, 2004) of the molecule. The U_{iso} values of the atoms in the central and outer portions of the citrate were constrained to be equal, and the U_{iso} values of the H atoms were constrained to be 1.3 times those of the atoms to which they are attached. Initial refinements suggested that K19 contained more electron density than the model and that the Rb22 site was occupied by less electron density. Accordingly, both cation sites were modeled as mixtures of K and Rb. The total occupation of each site was constrained to be 1.0, but the total K and Rb contents were not restrained, to provide a self-consistency check. The background was described by a 4-term

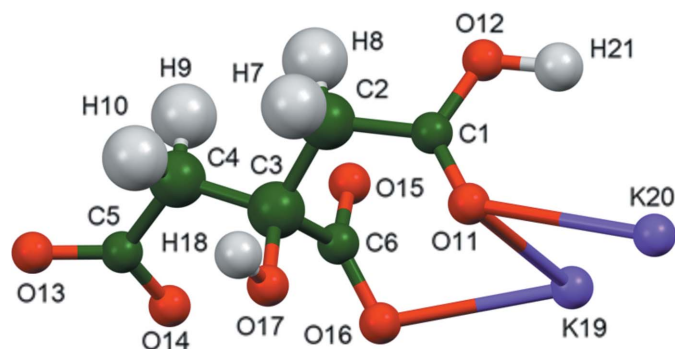


Figure 5
The asymmetric unit of the new polymorph of $\text{K}_2\text{HC}_6\text{H}_5\text{O}_7$, showing the atom numbering and 50% probability spheroids.

shifted Chebyshev polynomial, with a peak at 11.27° , to model the scattering from the glass capillary. The displacement coefficients of the atoms in the center of the citrate anion (C2/C3/C4/H7/H8/H9/H10) are quite large, suggesting disorder and/or approximate symmetry/unit cell.

Because density functional theory (DFT) techniques cannot accommodate disordered systems, two density functional geometry optimizations (with K19/Rb20 and Rb19/K20) were carried out using *CRYSTAL14* (Dovesi *et al.*, 2014). The basis sets for the H, C, N, and O atoms were those of Gatti *et al.* (1994), and the basis sets for K and Rb were those of Peintinger *et al.* (2013). The calculations were run on eight 2.1 GHz Xeon cores (each with 6 Gb RAM) of a 304-core Dell Linux cluster at IIT, using 8 k -points and the B3LYP functional, and took 3.2 and 1.8 d, respectively.

After storage for three years under ambient conditions, the sample was remeasured (kadu1880) and found to consist of a mixture of phases. One phase was identified as isostructural to ZZZQRO01, but with partial occupation of the K sites by Rb. The second phase was identified as isostructural to $\text{Rb}_2\text{HC}_6\text{H}_5\text{O}_7$ (Rammohan & Kaduk, 2017), but with partial occupation of the Rb sites by K. A Rietveld refinement (Fig. 4) showed that the sample consisted of 36.8 (4) wt% $\text{K}_{1.66}\text{Rb}_{0.34}\text{HC}_6\text{H}_5\text{O}_7$ and 63.2 (5) wt% $\text{K}_{0.29}\text{Rb}_{1.71}\text{HC}_6\text{H}_5\text{O}_7$.

3. Results and discussion

3.1. Dipotassium hydrogen citrate, $\text{K}_2\text{HC}_6\text{H}_5\text{O}_7$

The structure of a new polymorph of $\text{K}_2\text{HC}_6\text{H}_5\text{O}_7$ was solved and refined from laboratory powder data and optimized by DFT calculations (see *Experimental*) and is illu-

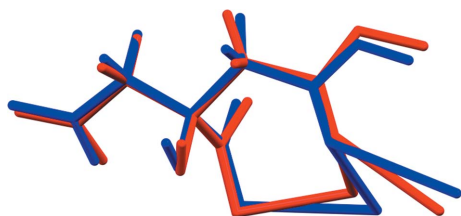


Figure 6
Comparison of the refined and optimized structures of the new polymorph of $\text{K}_2\text{HC}_6\text{H}_5\text{O}_7$. The refined structure is in red and the DFT-optimized structure is in blue. The r.m.s. Cartesian displacement is 0.332 Å.

strated in Fig. 5. The r.m.s. Cartesian displacement of the non-H citrate atoms in the Rietveld-refined and DFT-opti-

mized structures is 0.332 Å (Fig. 6). The agreement between the structures is at the upper end of the normal range for correct powder structures (van de Streek & Neumann, 2014). This discussion will emphasize the DFT-optimized structure.

All of the citrate bond lengths, bond angles, and torsion angles fall within the normal ranges indicated by a *Mercury/Mogul Geometry Check* (Macrae *et al.*, 2020). The citrate anion occurs in the *trans,trans* conformation (about C2–C3 and C3–C4), which is one of the two low-energy conformations of an isolated citrate anion (Rammohan & Kaduk, 2018). The central carboxylate group and the hydroxy group exhibit a small twist (O16–C6–C3–O17 torsion angle = -6.6°) from the normal planar arrangement. The Mulliken overlap populations indicate that the K–O bonds are ionic. K19 is seven-

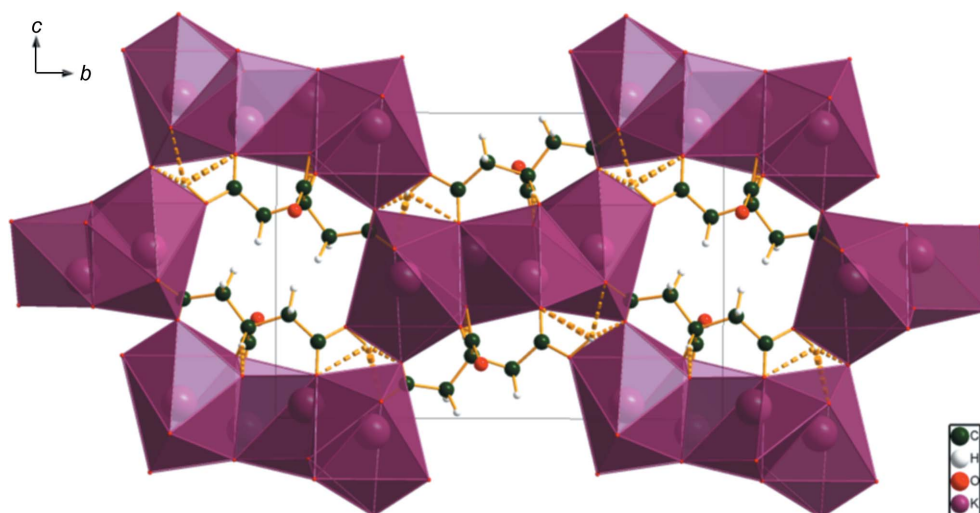


Figure 7
The crystal structure of the new polymorph of $\text{K}_2\text{HC}_6\text{H}_5\text{O}_7$, viewed down the a axis.

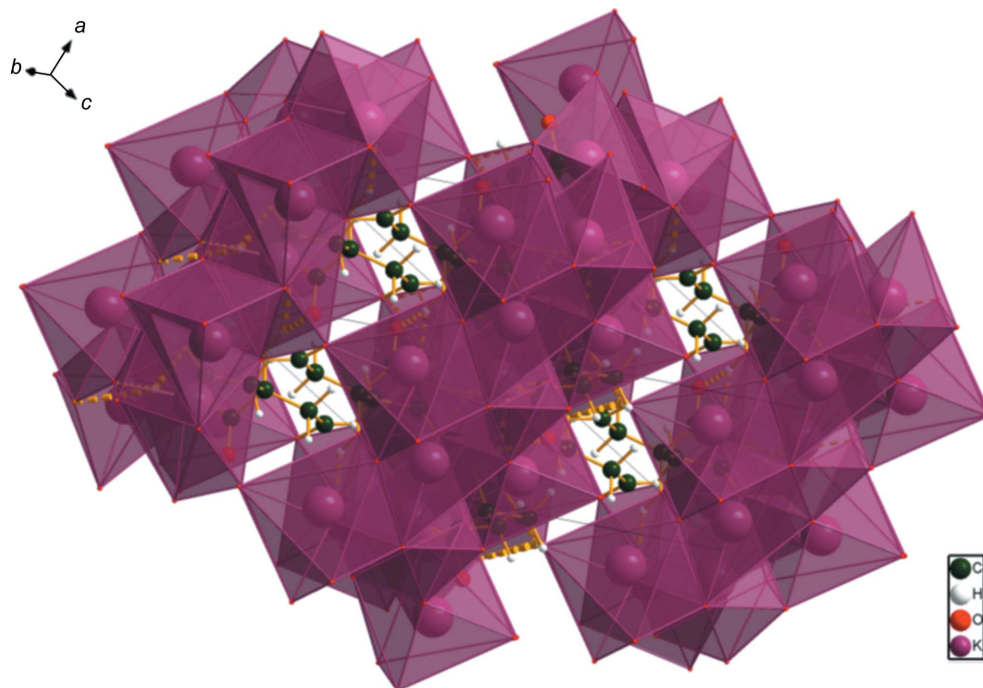


Figure 8
The crystal structure of the new polymorph of $\text{K}_2\text{HC}_6\text{H}_5\text{O}_7$, viewed down [111].

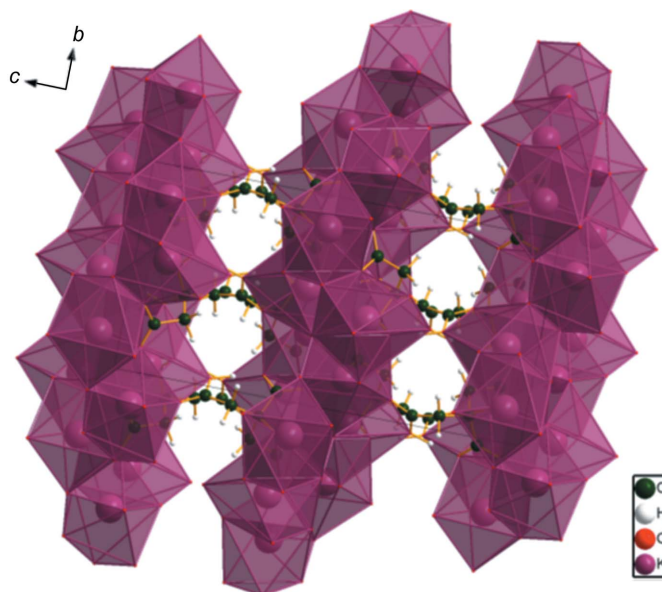


Figure 9
The crystal structure of the ZZZQRO01 polymorph of $K_2HC_6H_5O_7$, viewed down the a axis.

coordinate, with a bond valence sum of 1.13. K20 is eight-coordinate, with a bond valence sum of 1.04. Both coordination spheres are irregular.

The citrate anion triply chelates to K19 through the central carboxylate group, the terminal carboxylate O11 atom, and the hydroxy O17 group. The citrate double chelates to a second K19 atom through the central carboxylate O16 atom and the terminal carboxylate O11 atom. It chelates to K20 through the terminal carboxylate O13/O14 atoms and to a second K20 through the terminal carboxylate O14 atom and the central carboxylate O16 atom. All of the O atoms except O15 coordinate to at least one K^+ cation.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) method suggests that

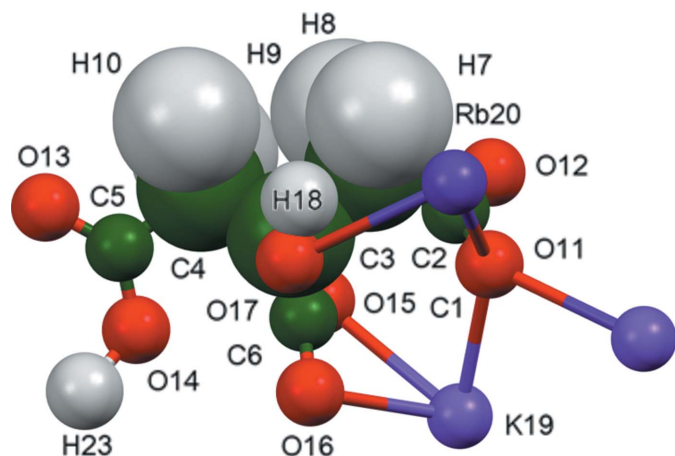


Figure 10
The asymmetric unit of $KRbHC_6H_5O_7$, showing the atom numbering and 50% probability spheroids.

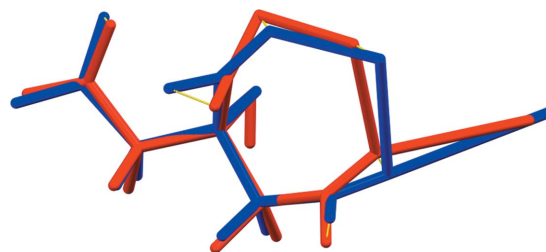


Figure 11
Comparison of the refined and optimized structures of $KRbHC_6H_5O_7$. The refined structure is in red and the DFT-optimized structure is in blue. The r.m.s. Cartesian displacement is 0.471 Å.

we might expect an elongated morphology for this new dipotassium hydrogen citrate, with {100} as the long axis. A second-order spherical harmonic preferred orientation model was used in the refinement. The texture index was only 1.004, indicating that the preferred orientation was not significant for this rotated capillary specimen.

The KO_7 and KO_8 coordination polyhedra share corners and edges to form a three-dimensional framework (Fig. 7), with channels parallel to the a axis and [111] (Fig. 8). The hydrophobic methylene groups face each other in the channels. The un-ionized carboxylic acid O12–H21 group forms a strong charge-assisted hydrogen bond to the ionized carboxylate O15 atom. The O17–H18 hydroxy group forms an intermolecular hydrogen bond to a different O15 atom. The energies of the hydrogen bonds were calculated using the correlation of Rammohan & Kaduk (2018).

In the structure of ZZZQRO01 (Zacharias & Glusker, 1993), the $K-O$ coordination polyhedra share edges and corners to form layers parallel to the ab plane (Fig. 9). There is a strong charge-assisted intramolecular hydrogen bond, and the hydroxy–carboxylate hydrogen bond is intermolecular. The *CRYSTAL14* energy of this new form of $K_2HC_6H_5O_7$ is -7834.807032 eV/cell. The energy of ZZZQRO01 is -7834.881818 eV/cell and is thus more stable by 0.074776 eV/cell, corresponding to 0.4 kcal mol⁻¹ per formula. The energy difference is within the expected error limits of such calculations, so the two polymorphs must be considered comparable in energy. The lower energy of ZZZQRO01 is consistent with conversion of the new polymorph to it over time.

The refined composition of 80% ZZZQRO01 $K_2HC_6H_5O_7$ and 20% $(NH_4)_2KC_6H_5O_7$ (Patel *et al.*, 2020) corresponds to a

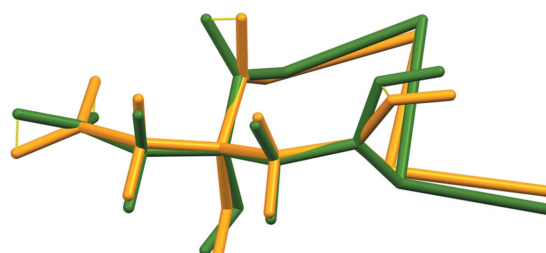


Figure 12
Comparison of the DFT-optimized structures of $KRbHC_6H_5O_7$ for K19/Rb20 (green) and Rb19/K20 (orange). The r.m.s. Cartesian difference is 0.447 Å.

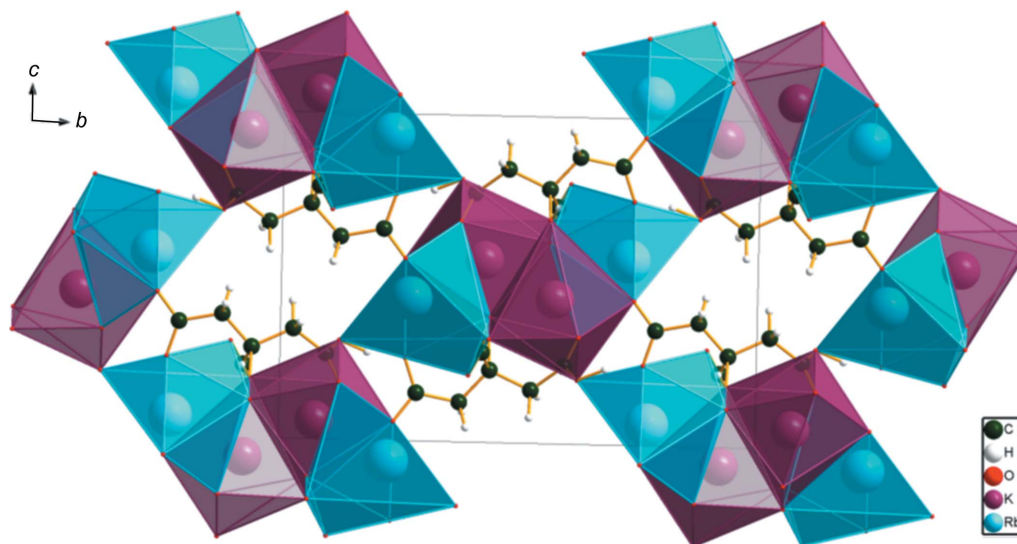


Figure 13
The crystal structure of $\text{KRbHC}_6\text{H}_5\text{O}_7$, viewed down the a axis.

K:N ratio of 4.4, instead of the expected ratio of 2.0. The difference suggests that some of the ammonium ions decomposed and/or were contained in an amorphous phase.

3.2. Potassium rubidium hydrogen citrate, $\text{KRbHC}_6\text{H}_5\text{O}_7$

The structure of $\text{KRbHC}_6\text{H}_5\text{O}_7$ was solved and refined from laboratory powder data and optimized by DFT calculations (see *Experimental*) and is illustrated in Fig. 10. The r.m.s. Cartesian displacement of the non-H citrate atoms in the Rietveld refined and DFT-optimized structures is 0.471 Å for K19 and 0.461 Å for Rb19 (Fig. 11). The two DFT-optimized

structures differ considerably (Fig. 12), suggesting that the large displacement coefficients in the Rietveld-refined structure result from disorder. The two optimized structures differ in energy by only 0.8 kcal mol⁻¹, which is within the expected uncertainties of such calculations. The similarities in energy and the size of the cations make disorder between K and Rb reasonable. The agreement between the structures is outside the normal range for correct powder structures (van de Streek & Neumann, 2014), but the disorder makes such a comparison more uncertain than usual. The refined cation site occupancies are K19/Rb20 = 0.71:0.29 and K21/Rb22 = 0.28:0.72. The absolute differences between the Rietveld cation positions

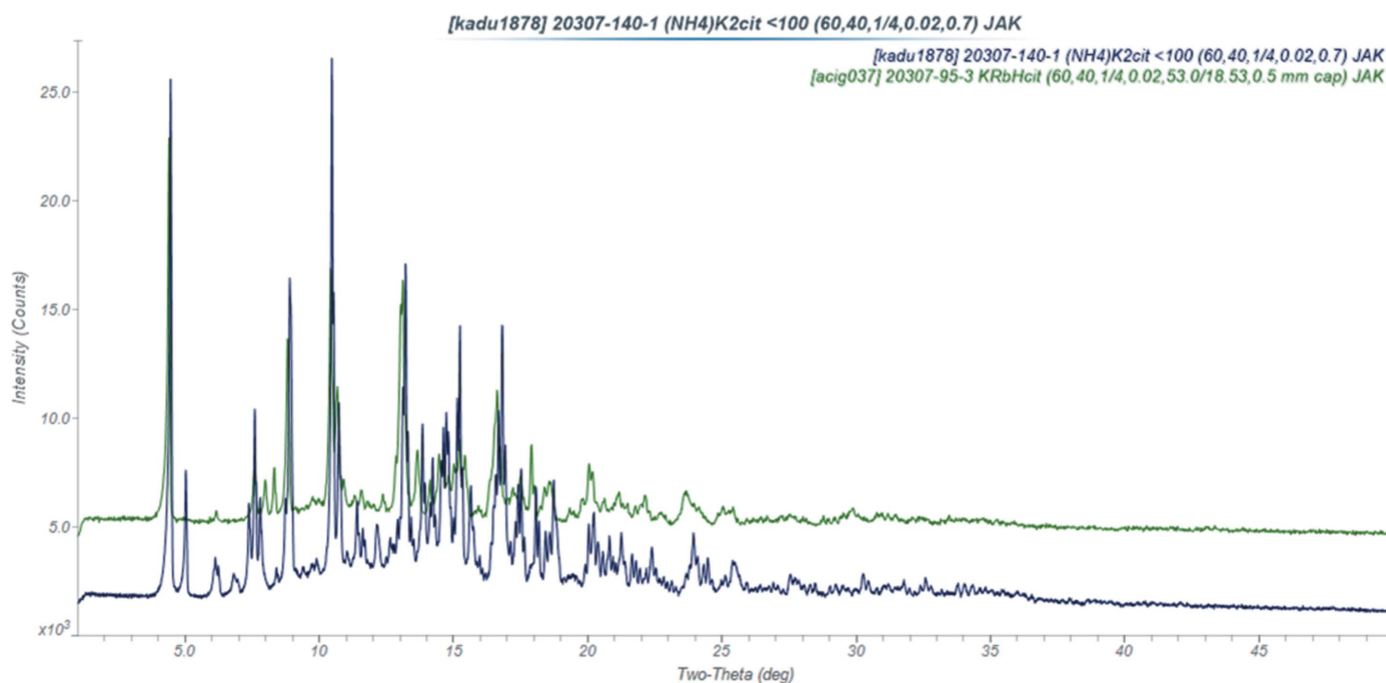


Figure 14
Comparison of the powder patterns of the new polymorph of $\text{K}_2\text{HC}_6\text{H}_5\text{O}_7$ and $\text{KRbHC}_6\text{H}_5\text{O}_7$.

and the K19/Rb20 DFT positions are 0.791 and 0.565 Å, respectively. The differences between the Rietveld positions and the Rb19/K20 DFT structure are 1.103 and 0.474 Å. This discussion will emphasize the DFT-optimized structure having the major occupancies of K19 and Rb20 (acig037_19_DFT in Table 1).

All of the citrate bond lengths, bond angles, and torsion angles fall within the normal ranges indicated by a *Mercury/Mogul Geometry Check* (Macrae *et al.*, 2020). The citrate anion occurs in the *trans,trans* conformation (about C2–C3 and C3–C4), which is one of the two low-energy conformations of an isolated citrate (Rammohan & Kaduk, 2018). The central carboxylate group and the hydroxy group exhibit a larger twist (O16–C6–C3–O17 torsion angle = -25.0°) from the normal planar arrangement. The Mulliken overlap populations indicate that the K–O and Rb–O bonds are ionic. K19 is eight-coordinate, with a bond valence sum of 1.15 and Rb22 is nine-coordinate, with a bond valence sum of 0.79. In the Rietveld-refined structure, the K19/Rb20 bond valence sums are 1.22 and 1.73, and the K21/Rb22 bond valence sums are 1.10 and 1.56.

The citrate anion triply chelates to Rb22 through the central carboxylate O15/O16 group and the terminal carboxylate O14 atom. The citrate double chelates to a K19 atom through the central carboxylate O16 atom and the hydroxy O17 group. It chelates to a second K19 atom through the terminal carboxylate O11 atom and the hydroxy O17 group. It triply chelates to a third K19 atom through the terminal carboxylate O11/O12 atoms and the central carboxylate O16 atom.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) method suggests that we might expect an elongated morphology for potassium

rubidium hydrogen citrate. A preferred orientation model was not necessary, indicating that the preferred orientation was not significant for this rotated capillary specimen.

KO₈ and RbO₉ coordination polyhedra share corners and edges to form a three-dimensional framework (Fig. 13) with channels parallel to the *a* axis. The hydrophobic methylene groups face each other in the channels. The un-ionized carboxylic acid O12–H23 group forms a strong charge-assisted hydrogen bond to the ionized carboxylate O14 group. The hydroxy O17–H18 group forms an intermolecular hydrogen bond to the central carboxylate O15 group. The energies of the hydrogen bonds were calculated using the correlation of Rammohan & Kaduk (2018). The DFT energies suggest that the K19/Rb20 structure is lower in energy by 0.2 kcal mol⁻¹; this difference is within the expected uncertainty in such calculations, so the two structures must be considered equivalent in energy.

The powder patterns of the new polymorph of K₂HC₆H₅O₇ and KRbHC₆H₅O₇ are similar (Fig. 14), reflecting the similarity in the structures (Figs. 7 and 13). After storage for three years under ambient conditions, KRbHC₆H₅O₇ changed into a mixture of 36.8 (4) wt% K_{1.66}Rb_{0.34}HC₆H₅O₇ and 63.2 (5) wt% K_{0.29}Rb_{1.71}HC₆H₅O₇ (Fig. 15). This mixture of these compositions corresponds to a Rb/K ratio of 1.32, compared to the expected ratio of 1.00. The difference suggests that the refined site occupancies might not be as accurate as expected, and/or that some amorphous phase is also present.

Acknowledgements

We thank North Central College for allowing us the space and resources to pursue this research project. We also thank the

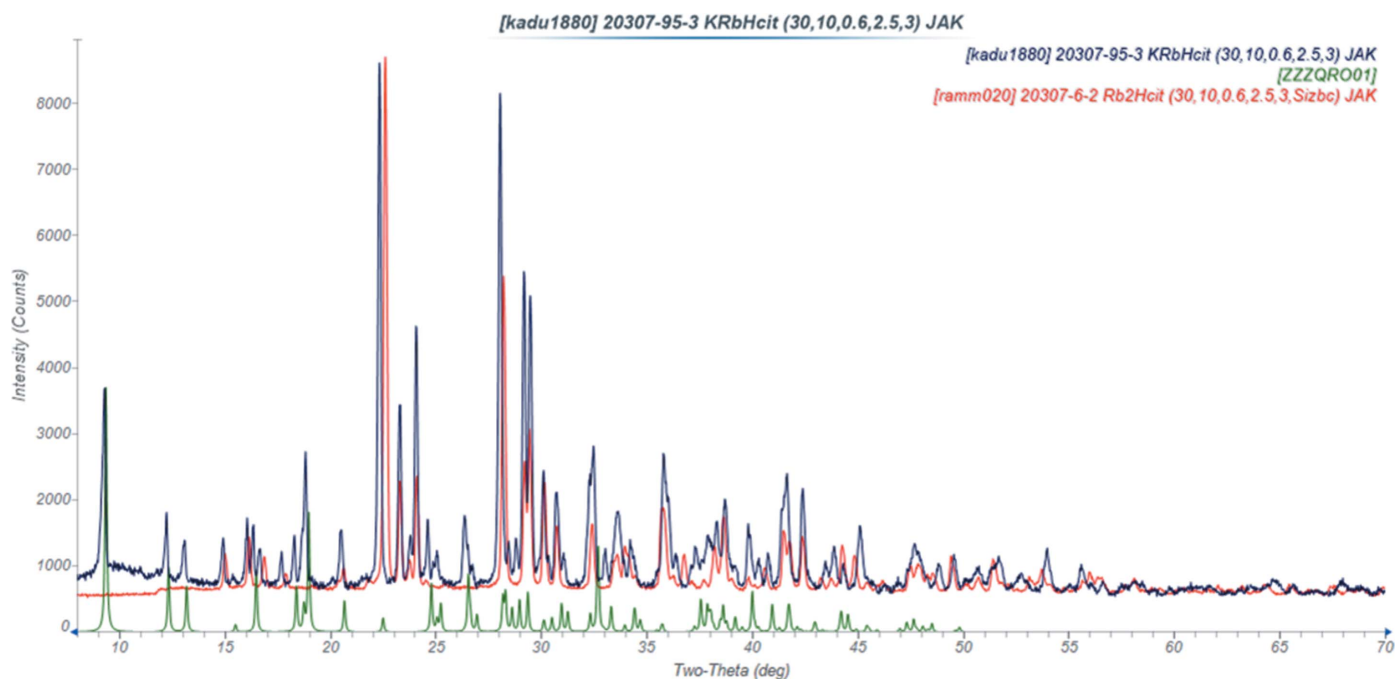


Figure 15
Comparison of the powder pattern of decomposed KRbHC₆H₅O₇ (black) to those of K₂HC₆H₅O₇ (ZZZQRO01, green) and Rb₂HC₆H₅O₇ (red).

Illinois Mathematics and Science Academy for offering us the opportunity to work on this project. We thank Andrey Rogachev for the use of computing resources at the Illinois Institute of Technology.

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supporting information

Acta Cryst. (2020). C76, 706-715 [https://doi.org/10.1107/S2053229620008281]

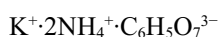
Crystal structures of two isostructural compounds: a second polymorph of dipotassium hydrogen citrate, $K_2HC_6H_5O_7$, and potassium rubidium hydrogen citrate, $KRbHC_6H_5O_7$

Diana Gonzalez, Joseph T. Golab, Andrew J. Cigler and James A. Kaduk

Computing details

Diammonium potassium citrate (kadu1878_phase_1)

Crystal data



$M_r = 264.27$

Monoclinic, $P2_1/c$

$a = 5.952$ (5) Å

$b = 13.261$ (9) Å

$c = 13.266$ (7) Å

$\beta = 92.88$ (5)°

$V = 1045.8$ (8) Å³

$Z = 4$

$D_x = 1.679$ Mg m⁻³

$T = 304$ K

cylinder, 12 × 0.7 mm

Data collection

PANalytical Empyrean
diffractometer

Specimen mounting: glass capillary

Data collection mode: transmission

Scan method: step

Refinement

Profile function: Crystallite size in microns with
"isotropic" model: parameters: Size, G/L mix
1.000, 1.000, Microstrain, "isotropic" model
(10⁶ * delta Q/Q) parameters: Mustrain, G/L
mix 7.0(15)e3, 1.000,

58 restraints

$(\Delta/\sigma)_{\max} < 0.001$

Preferred orientation correction: Simple

spherical harmonic correction Order = 2

Coefficients: 1:0:C(2,-2) = 0.30(8); 1:0:C(2,0) =
-0.39(5); 1:0:C(2,2) = 0.11(5)

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.88640	0.03730	0.85830	0.012*
C2	0.75700	0.08120	0.94220	0.032*
C3	0.79310	0.19420	0.96080	0.032*
C4	0.66920	0.22650	1.05270	0.032*
C5	0.65560	0.33900	1.07210	0.012*
C6	0.70430	0.25400	0.86790	0.012*
H7	0.77670	0.04320	0.99960	0.042*
H8	0.57450	0.06940	0.92620	0.042*
H9	0.50370	0.20020	1.04630	0.042*

H10	0.71580	0.19530	1.10950	0.042*
O12	0.80800	-0.03900	0.81450	0.012*
O13	0.47350	0.37350	1.09710	0.012*
O14	0.82650	0.39030	1.06390	0.012*
O15	0.49800	0.25950	0.85270	0.012*
O16	0.84460	0.29290	0.81590	0.012*
O17	1.02580	0.21300	0.97840	0.012*
O11	1.06080	0.08060	0.83620	0.012*
H18	1.08650	0.16790	0.93840	0.015*
N19	0.82540	0.59910	0.07430	0.030*
N20	0.36780	0.97550	0.74130	0.030*
K21	0.84140	0.75840	0.30680	0.103*
H22	0.93790	0.61560	0.05380	0.039*
H23	0.90790	0.60060	0.13800	0.039*
H24	0.69960	0.62600	0.08460	0.039*
H25	0.79170	0.52180	0.07060	0.039*
H26	0.30840	0.91370	0.72030	0.039*
H27	0.26010	1.01030	0.77260	0.039*
H28	0.38690	1.01050	0.67920	0.039*
H29	0.50180	0.97670	0.76900	0.039*

Geometric parameters (Å, °)

C1—C2	1.5024	O15—K21 ^{iv}	2.8609
C1—O12	1.2458	O16—C6	1.2236
C1—O11	1.2344	O16—K21 ^v	2.6289
C2—C1	1.5024	O17—C3	1.4151
C2—C3	1.5321	O17—H18	0.8883
C2—H7	0.9158	O17—K21 ⁱⁱⁱ	2.9802
C2—H8	1.1074	O11—C1	1.2344
C3—C2	1.5321	O11—K21 ^v	2.9338
C3—C4	1.5179	H18—O17	0.8883
C3—C6	1.5368	N19—H22	0.7671
C3—O17	1.4151	N19—H23	0.9560
C4—C3	1.5179	N19—H24	0.8467
C4—C5	1.5168	N19—H25	1.0452
C4—H9	1.0444	N20—H26	0.9297
C4—H10	0.8916	N20—H27	0.9072
C5—C4	1.5168	N20—H28	0.9573
C5—O13	1.237	N20—H29	0.8614
C5—O14	1.2331	K21—O12 ^{vi}	2.9184
C6—C3	1.5368	K21—O13 ^{vii}	2.7764
C6—O15	1.2364	K21—O14 ^{viii}	3.0933
C6—O16	1.2236	K21—O15 ^{iv}	2.8609
H7—C2	0.9158	K21—O16 ^v	2.6289
H8—C2	1.1074	K21—O17 ^{viii}	2.9802
H9—C4	1.0444	K21—O11 ^v	2.9338
H9—H10	1.4814	H22—N19	0.7671

H10—C4	0.8916	H23—N19	0.9560
H10—H9	1.4814	H23—H22	1.1573
O12—C1	1.2458	H24—N19	0.8467
O12—K21 ⁱ	2.9184	H25—N19	1.0452
O13—C5	1.237	H26—N20	0.9297
O13—K21 ⁱⁱ	2.7764	H27—O11 ^{ix}	1.7575
O14—C5	1.2331	H27—N20	0.9072
O14—K21 ⁱⁱⁱ	3.0933	H28—N20	0.9573
O15—C6	1.2364	H29—N20	0.8614
C2—C1—O12	117.797	C4—C5—O14	118.45
C2—C1—O11	117.66	O13—C5—O14	123.876
O12—C1—O11	124.526	C3—C6—O15	117.383
C1—C2—C3	115.174	C3—C6—O16	116.92
C1—C2—H7	110.723	O15—C6—O16	125.694
C3—C2—H7	113.156	C5—O13—K21 ⁱⁱ	122.724
C1—C2—H8	109.735	C6—O16—K21 ^v	140.044
C3—C2—H8	107.299	C3—O17—H18	101.94
H7—C2—H8	99.498	H22—N19—H23	83.613
C2—C3—C4	109.603	H22—N19—H24	137.036
C2—C3—C6	109.618	H23—N19—H24	105.526
C4—C3—C6	109.703	H22—N19—H25	115.591
C2—C3—O17	109.091	H23—N19—H25	98.67
C4—C3—O17	109.192	H24—N19—H25	104.573
C6—C3—O17	109.617	H26—N20—H27	108.588
C3—C4—C5	116.492	H26—N20—H28	103.256
C3—C4—H9	109.559	H27—N20—H28	105.335
C5—C4—H9	106.505	H26—N20—H29	118.304
C3—C4—H10	113.88	H27—N20—H29	117.111
C5—C4—H10	109.27	H28—N20—H29	102.166
H9—C4—H10	99.545	O13 ^{vii} —K21—O16 ^v	160.723
C4—C5—O13	117.66		

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

Dipotassium hydrogen citrate (kadu1878_phase_0)

Crystal data

$2\text{K}^+\cdot\text{C}_6\text{H}_6\text{O}_7^{2-}$

$M_r = 268.3$

Monoclinic, $P2_1/c$

$a = 5.3473$ (3) Å

$b = 16.1428$ (10) Å

$c = 11.0103$ (8) Å

$\beta = 91.438$ (4)°

$V = 950.12$ (13) Å³

$Z = 4$

$D_x = 1.876$ Mg m⁻³

$T = 304$ K

cylinder, 12×0.7 mm

Data collection

PANalytical Empyrean
diffractometer

Specimen mounting: glass capillary

Data collection mode: transmission

Scan method: step

Refinement

Profile function: Crystallite size in microns with "isotropic" model: parameters: Size, G/L mix 1.000, 1.000, Microstrain, "isotropic" model ($10^6 \cdot \Delta Q/Q$) parameters: Mustrain, G/L mix 6.0(10)e2, 1.000, 58 restraints

$(\Delta/\sigma)_{\max} < 0.001$
Preferred orientation correction: Simple spherical harmonic correction Order = 2
Coefficients: 0:0:C(2,-2) = -0.030(13); 0:0:C(2,0) = 0.012(14); 0:0:C(2,2) = -0.135(13)

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	0.286 (3)	0.5926 (7)	0.2244 (11)	0.036 (2)*
C2	0.175 (3)	0.5173 (8)	0.1555 (12)	0.071 (7)*
C3	0.2345 (17)	0.4304 (6)	0.2106 (9)	0.071*
C4	0.225 (3)	0.3634 (8)	0.1069 (12)	0.071*
C5	0.330 (3)	0.2784 (7)	0.1405 (17)	0.0362*
C6	0.4578 (18)	0.4341 (7)	0.2563 (8)	0.0362*
H7	-0.05249	0.52515	0.17538	0.093*
H8	0.18896	0.51333	0.05945	0.093*
H9	0.29453	0.38533	0.03244	0.093*
H10	-0.01458	0.35783	0.09400	0.093*
O11	0.216 (3)	0.5972 (8)	0.3378 (12)	0.0362*
O12	0.357 (3)	0.6531 (10)	0.1529 (15)	0.0362*
O13	0.221 (3)	0.2212 (9)	0.0950 (14)	0.0362*
O14	0.530 (3)	0.2796 (9)	0.1998 (15)	0.0362*
O15	0.605 (2)	0.4715 (10)	0.1824 (13)	0.0362*
O16	0.456 (3)	0.4299 (10)	0.3849 (12)	0.0362*
O17	0.071 (2)	0.4085 (9)	0.3156 (14)	0.0362*
H18	-0.03380	0.42830	0.28050	0.0471*
K19	0.7605 (13)	0.9456 (5)	0.8919 (6)	0.039 (2)*
K20	0.2440 (12)	0.6955 (4)	0.5320 (6)	0.0392*
H21	0.44500	0.71980	0.21210	0.050*

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

C1—C2	1.542 (9)	O13—C5	1.195 (12)
C1—O11	1.315 (13)	O13—K19 ⁱⁱⁱ	2.698 (14)
C1—O12	1.316 (12)	O13—K20 ^{iv}	2.853 (14)
C2—C1	1.542 (9)	O14—C5	1.240 (13)
C2—C3	1.558 (9)	O14—H21 ^v	1.373 (15)
C2—H7	1.250 (16)	O15—C6	1.294 (12)
C2—H8	1.064 (14)	O15—K19 ⁱⁱ	2.777 (16)
C3—C2	1.558 (9)	O16—C6	1.418 (13)
C3—C4	1.573 (9)	O16—K19 ^{vi}	2.753 (14)
C3—C6	1.285 (8)	O16—K19 ⁱⁱ	2.587 (15)
C3—O17	1.509 (12)	O16—K20 ⁱⁱⁱ	2.728 (17)
C4—C3	1.573 (9)	O17—C3	1.509 (12)
C4—C5	1.523 (9)	O17—H18	0.744 (14)
C4—H9	0.975 (16)	O17—K19 ⁱ	3.013 (15)

C4—H10	1.289 (17)	O17—K20 ^{vii}	2.935 (14)
C5—C4	1.523 (9)	H18—O17	0.744 (14)
C5—O13	1.195 (12)	H18—K19 ⁱ	2.632 (7)
C5—O14	1.240 (13)	K19—O11 ^{viii}	3.038 (17)
C6—C3	1.285 (8)	K19—O11 ^{ix}	2.615 (17)
C6—O15	1.294 (12)	K19—O13 ⁱⁱⁱ	2.698 (14)
C6—O16	1.418 (13)	K19—O15 ^{viii}	2.777 (16)
H7—C2	1.250 (16)	K19—O16 ^x	2.753 (14)
H8—C2	1.064 (14)	K19—O16 ^{viii}	2.587 (15)
H9—C4	0.975 (16)	K19—O17 ^{ix}	3.013 (15)
H10—C4	1.289 (17)	K20—O11	2.663 (14)
O11—C1	1.315 (13)	K20—O12 ^{viii}	2.841 (15)
O11—K19 ⁱ	2.615 (17)	K20—O13 ^{xi}	2.853 (14)
O11—K19 ⁱⁱ	3.038 (17)	K20—O16 ⁱⁱⁱ	2.728 (17)
O11—K20	2.663 (14)	K20—O17 ^{vii}	2.935 (14)
O12—C1	1.316 (12)	H21—O12	1.338 (16)
O12—K20 ⁱⁱ	2.841 (15)	H21—O14 ^{xii}	1.373 (15)
O12—H21	1.338 (16)		
C2—C1—O11	113.4 (5)	K19 ⁱ —O11—K20	90.5 (6)
C2—C1—O12	113.8 (5)	C1—O12—H21	114.1 (10)
O11—C1—O12	128.3 (5)	C5—O13—K19 ⁱⁱⁱ	137.1 (8)
C1—C2—C3	116.5 (5)	C5—O14—H21 ^v	115.4 (12)
C1—C2—H7	101.4 (10)	C6—O15—K19 ⁱⁱ	82.9 (6)
C3—C2—H7	102.2 (9)	C6—O16—K19 ^{vi}	154.3 (9)
C1—C2—H8	120.1 (11)	C6—O16—K19 ⁱⁱ	88.4 (7)
C3—C2—H8	108.3 (9)	K19 ^{vi} —O16—K19 ⁱⁱ	100.4 (5)
H7—C2—H8	105.7 (9)	C6—O16—K20 ⁱⁱⁱ	110.6 (9)
C2—C3—C6	106.7 (4)	K19 ^{vi} —O16—K20 ⁱⁱⁱ	91.5 (5)
C2—C3—O17	113.2 (5)	K19 ⁱⁱ —O16—K20 ⁱⁱⁱ	101.6 (5)
C6—C3—O17	105.2 (5)	C3—O17—H18	86.9 (12)
C5—C4—H9	112.7 (14)	O11 ^{ix} —K19—O13 ⁱⁱⁱ	73.5 (5)
C5—C4—H10	108.8 (10)	O11 ^{ix} —K19—O15 ^{viii}	101.5 (5)
H9—C4—H10	109.3 (10)	O13 ⁱⁱⁱ —K19—O15 ^{viii}	122.3 (6)
C4—C5—O13	115.0 (4)	O11 ^{ix} —K19—O16 ^x	126.4 (5)
C4—C5—O14	114.9 (5)	O13 ⁱⁱⁱ —K19—O16 ^x	82.9 (5)
O13—C5—O14	129.5 (5)	O15 ^{viii} —K19—O16 ^x	131.4 (5)
C3—C6—O15	110.3 (5)	O11 ^{ix} —K19—O16 ^{viii}	142.0 (5)
C3—C6—O16	111.0 (5)	O13 ⁱⁱⁱ —K19—O16 ^{viii}	143.0 (6)
O15—C6—O16	132.1 (5)	O15 ^{viii} —K19—O16 ^{viii}	54.9 (4)
C1—O11—K19 ⁱ	119.4 (9)	O16 ^x —K19—O16 ^{viii}	79.6 (5)
C1—O11—K20	141.7 (8)	O11—K20—O16 ⁱⁱⁱ	81.3 (5)

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

(kadu1878_DFT)

*Crystal data*2K⁺·C₆H₆O₇²⁻ $M_r = 268.3$ Monoclinic, $P2_1/C$ $a = 5.3473 \text{ \AA}$ $b = 16.1429 \text{ \AA}$ $c = 11.0103 \text{ \AA}$ $\beta = 91.4375^\circ$ $V = 950.12 \text{ \AA}^3$ $Z = 4$ *Data collection*

DFT 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.27542	0.59432	0.25102	0.03650*
C2	0.19377	0.52852	0.16233	0.06900*
C3	0.25141	0.43837	0.20473	0.06900*
C4	0.21888	0.38139	0.09382	0.06900*
C5	0.30269	0.29250	0.11215	0.03650*
C6	0.51984	0.43856	0.25349	0.03650*
H7	-0.00789	0.53398	0.14811	0.09000*
H8	0.28366	0.53981	0.07651	0.09000*
H9	0.32770	0.40568	0.01905	0.09000*
H10	0.02298	0.38205	0.06465	0.09000*
O11	0.23676	0.59182	0.36137	0.03650*
O12	0.39428	0.65679	0.20151	0.03650*
O13	0.19259	0.23603	0.05356	0.03650*
O14	0.49576	0.27994	0.18085	0.03650*
O15	0.68164	0.45872	0.17766	0.03650*
O16	0.56672	0.42548	0.36494	0.03650*
O17	0.09030	0.41267	0.29901	0.03650*
H18	-0.07973	0.42494	0.26656	0.04740*
K19	0.76601	0.93180	0.95999	0.03880*
K20	0.26419	0.71591	0.54539	0.03880*
H21	0.44724	0.70549	0.26426	0.05000*

Bond lengths (\AA)

C1—C2	1.500	C4—C5	1.515
C1—O11	1.238	C4—H9	1.093
C1—O12	1.317	C4—H10	1.088
C2—C3	1.557	C5—O13	1.255
C2—H7	1.089	C5—O14	1.281
C2—H8	1.086	C6—O15	1.260
C3—C4	1.535	C6—O16	1.264
C3—C6	1.520	O12—H21	1.080
C3—O17	1.427	O17—H18	0.989

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O12—H21 \cdots O14	1.080	1.376	2.437	165.6
O17—H18 \cdots O15	0.989	1.680	2.639	162.6

Diammonium potassium citrate (kadu1860_phase_1)

Crystal data

$K^+ \cdot 2NH_4^+ \cdot C_6H_5O_7^{3-}$

$M_r = 264.27$

Monoclinic, $P2_1/c$

$a = 5.993$ (2) Å

$b = 13.260$ (3) Å

$c = 13.329$ (4) Å

$\beta = 93.35$ (2)°

$V = 1057.3$ (4) Å³

$Z = 4$

$D_x = 1.660$ Mg m⁻³

$T = 300$ K

flat_sheet, 25 × 25 mm

Data collection

Bruker D2 Phaser
diffractometer

Specimen mounting: PMMA holder with
Kapton film

Data collection mode: reflection

Scan method: step

Refinement

Profile function: Crystallite size in microns with
"isotropic" model: parameters: Size, G/L mix
1.000, 1.000, Microstrain, "isotropic" model
(10⁶ * delta Q/Q) parameters: Mustrain, G/L
mix 4.6(6)e3, 1.000,

0 restraints

Preferred orientation correction: March-Dollase
correction coef. = 1.000 axis = [0, 0, 1]

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

	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C1	0.87318	0.03565	0.86687	0.012*
C2	0.72599	0.08314	0.94344	0.036*
C3	0.76903	0.19496	0.96828	0.036*
C4	0.63480	0.22315	1.05833	0.036*
C5	0.63310	0.33416	1.08863	0.012*
C6	0.68765	0.25946	0.87689	0.012*
H7	0.75607	0.03931	1.01180	0.046*
H8	0.55188	0.07301	0.91973	0.046*
H9	0.46410	0.19855	1.04385	0.046*
H10	0.70341	0.18054	1.12249	0.046*
O12	0.80347	-0.04262	0.82266	0.012*
O13	0.44391	0.37143	1.10579	0.012*
O14	0.81343	0.38194	1.09793	0.012*
O15	0.48252	0.26029	0.85377	0.012*
O16	0.83050	0.30870	0.83032	0.012*
O17	0.99968	0.21047	0.99448	0.012*

O11	1.06265	0.07558	0.85523	0.012*
H18	1.07924	0.17143	0.94538	0.016*
N20	0.37168	0.98204	0.74458	0.040*
K21	0.86217	0.75540	0.30100	0.040*
H22	0.78800	0.60364	0.00308	0.052*
H23	0.95796	0.62600	0.10404	0.052*
N19	0.81121	0.59136	1.07827	0.040*
H24	0.68536	0.63002	0.11046	0.052*
H25	0.80797	0.51452	0.09426	0.052*
H26	0.30306	0.91502	0.71752	0.052*
H27	0.25619	1.01594	0.78942	0.052*
H28	0.40466	1.03125	0.68656	0.052*
H29	0.52233	0.96762	0.78341	0.052*

Geometric parameters (Å, °)

C1—C2	1.5236	O16—C6	1.2675
C1—O12	1.2531	O16—K21 ^v	2.7486
C1—O11	1.2705	O17—C3	1.4207
C2—C1	1.5236	O17—H18	0.9798
C2—C3	1.5378	O17—K21 ⁱⁱⁱ	2.8649
C2—H7	1.0869	O11—C1	1.2705
C2—H8	1.0808	O11—K21 ^v	3.1100
C3—C2	1.5378	H18—O17	0.9798
C3—C4	1.5298	N20—H26	1.0352
C3—C6	1.5439	N20—H27	1.0424
C3—O17	1.4207	N20—H28	1.0396
C4—C3	1.5298	N20—H29	1.0319
C4—C5	1.5266	K21—O12 ^{vi}	2.8600
C4—H9	1.0803	K21—O13 ^{vii}	2.7462
C4—H10	1.0856	K21—O14 ^{viii}	2.8466
C5—C4	1.5266	K21—O15 ^{iv}	2.8403
C5—O13	1.2698	K21—O16 ^v	2.7486
C5—O14	1.2526	K21—O17 ^{viii}	2.8649
C6—C3	1.5439	K21—O11 ^v	3.1100
C6—O15	1.2498	H22—N19 ^{ix}	1.0168
C6—O16	1.2675	H23—O16 ^v	1.7299
H7—C2	1.0869	H23—N19 ^{ix}	1.0330
H8—C2	1.0808	N19—H22 ^x	1.0168
H9—C4	1.0803	N19—H23 ^x	1.0330
H10—C4	1.0856	N19—H24 ^x	1.0264
O12—C1	1.2531	N19—H25 ^x	1.0414
O12—K21 ⁱ	2.8600	H24—N19 ^{ix}	1.0264
O13—C5	1.2698	H25—N19 ^{ix}	1.0414
O13—K21 ⁱⁱⁱ	2.7462	H26—N20	1.0352
O14—C5	1.2526	H27—O11 ^{xi}	1.6909
O14—K21 ⁱⁱⁱ	2.8466	H27—N20	1.0424
O15—C6	1.2498	H28—N20	1.0396

O15—K21 ^{iv}	2.8403	H29—N20	1.0319
C2—C1—O12	117.851	C4—C5—O14	119.659
C2—C1—O11	117.779	O13—C5—O14	124.027
O12—C1—O11	124.298	C3—C6—O15	117.4
C1—C2—C3	116.51	C3—C6—O16	118.718
C1—C2—H7	105.45	O15—C6—O16	123.88
C3—C2—H7	108.518	C5—O13—K21 ⁱⁱ	119.371
C1—C2—H8	109.852	C6—O16—K21 ^v	130.499
C3—C2—H8	109.251	C3—O17—H18	105.293
H7—C2—H8	106.777	H26—N20—H27	107.844
C2—C3—C4	108.376	H26—N20—H28	111.629
C2—C3—C6	108.845	H27—N20—H28	108.499
C4—C3—C6	109.106	H26—N20—H29	109.496
C2—C3—O17	109.94	H27—N20—H29	112.524
C4—C3—O17	108.689	H28—N20—H29	106.894
C6—C3—O17	111.818	O13 ^{vii} —K21—O16 ^v	162.076
C3—C4—C5	117.073	H22 ^x —N19—H23 ^x	108.739
C3—C4—H9	108.779	H22 ^x —N19—H24 ^x	105.694
C5—C4—H9	108.513	H23 ^x —N19—H24 ^x	105.724
C3—C4—H10	107.371	H22 ^x —N19—H25 ^x	110.811
C5—C4—H10	107.55	H23 ^x —N19—H25 ^x	113.124
H9—C4—H10	107.137	H24 ^x —N19—H25 ^x	112.345
C4—C5—O13	116.282		

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

Dipotassium hydrogen citrate (kadu1860_phase_0)

Crystal data

$2\text{K}^+\cdot\text{C}_6\text{H}_6\text{O}_7^{2-}$

$M_r = 268.3$

Monoclinic, $C2/c$

$a = 10.7735$ (6) Å

$b = 9.6647$ (5) Å

$c = 18.9330$ (8) Å

$\beta = 90.045$ (6)°

$V = 1971.4$ (2) Å³

$Z = 8$

$D_x = 1.808$ Mg m⁻³

$T = 300$ K

flat_sheet, 25 × 25 mm

Data collection

Bruker D2 Phaser
diffractometer

Specimen mounting: PMMA holder with
Kapton film

Data collection mode: reflection

Scan method: step

Refinement

Profile function: Crystallite size in microns with "isotropic" model: parameters: Size, G/L mix 1.000, 1.000, Microstrain, "isotropic" model ($10^6 \cdot \Delta Q/Q$) parameters: Mustrain, G/L mix 2.23(8)e3, 1.000, 0 restraints

Preferred orientation correction: Simple spherical harmonic correction Order = 6
Coefficients: 0:0:C(2,-2) = -0.430(30);
0:0:C(2,0) = 0.457(31); 0:0:C(2,2) = -0.12(4);
0:0:C(4,-4) = 0.41(6); 0:0:C(4,-2) = -0.65(7);
0:0:C(4,0) = 0.20(6); 0:0:C(4,2) = 0.04(5);
0:0:C(4,4) = 1.21(7); 0:0:C(6,-6) = -0.96(8);
0:0:C(6,-4) = 0.22(8); 0:0:C(6,-2) = -0.17(9);
0:0:C(6,0) = -1.12(9); 0:0:C(6,2) = -0.97(11);
0:0:C(6,4) = 0.26(8); 0:0:C(6,6) = -0.27(10)

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	0.25901	0.04541	0.22942	0.030*
C2	0.18032	0.06770	0.29481	0.030*
C3	0.24734	0.09830	0.36529	0.030*
C4	0.84937	0.70884	0.35433	0.030*
C5	0.90431	0.75739	0.42438	0.030*
C6	0.30314	-0.03537	0.39758	0.030*
H7	0.12145	-0.02382	0.30156	0.039*
H8	0.11908	0.15470	0.28379	0.039*
H9	-0.19189	-0.20405	0.32631	0.039*
H10	-0.07615	-0.33414	0.32205	0.039*
O11	0.23110	0.09838	0.17278	0.030*
O12	0.35671	-0.03752	0.23393	0.030*
O13	0.87503	0.87696	0.44664	0.030*
O14	0.97406	0.67389	0.45700	0.030*
O15	0.88346	0.40040	0.36001	0.030*
O16	0.26835	0.92611	0.45712	0.030*
O17	0.15267	0.15276	0.41038	0.030*
H18	0.16110	0.11919	0.45951	0.039*
K19	0.10153	0.15846	0.05435	0.030*
K20	-0.38269	-0.04931	0.42269	0.030*
H21	0.37493	-0.06869	0.28630	0.039*

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

C1—C2	1.5164	O14—C5	1.2636
C1—O11	1.2255	O14—K19 ^{viii}	2.8115
C1—O12	1.3257	O14—K20 ^{ix}	2.7311
C2—C1	1.5164	O14—K20 ^x	2.7587
C2—C3	1.5453	O15—C6 ⁱⁱ	1.2810
C2—H7	1.0959	O15—K19 ^{xi}	2.8500
C2—H8	1.0889	O15—K20 ^{ix}	2.8262
C3—C2	1.5453	O15—H21 ⁱⁱ	1.4301
C3—C4 ⁱ	1.5470	O16—C6 ^{xii}	1.2452
C3—C6	1.5503	O16—K19 ^{xiii}	2.7004
C3—O17	1.4309	O16—K19 ^{xiv}	2.9503

C4—C3 ⁱⁱ	1.5470	O16—K20 ^{xv}	2.8476
C4—C5	1.5259	O17—C3	1.4309
C4—H9 ⁱⁱⁱ	1.0897	O17—H18	0.9893
C4—H10 ⁱⁱⁱ	1.0912	O17—K19 ^{vi}	2.8199
C5—C4	1.5259	O17—K20 ⁱⁱ	2.9139
C5—O13	1.2700	H18—O17	0.9893
C5—O14	1.2636	K19—O11	2.7034
C6—C3	1.5503	K19—O13 ^{xvi}	2.7324
C6—O15 ⁱ	1.2810	K19—O14 ^{xvii}	2.8115
C6—O16 ^{iv}	1.2452	K19—O15 ^{xi}	2.8500
H7—C2	1.0959	K19—O16 ^{xviii}	2.7004
H8—C2	1.0889	K19—O16 ^{xix}	2.9503
H9—C4 ^v	1.0897	K19—O17 ^{vi}	2.8199
H10—C4 ^v	1.0912	K20—O11 ^{vi}	2.8243
O11—C1	1.2255	K20—O12 ^{vi}	2.9809
O11—K19	2.7034	K20—O13 ^v	2.9018
O11—K20 ^{vi}	2.8243	K20—O14 ^{xx}	2.7311
O12—C1	1.3257	K20—O14 ^x	2.7587
O12—K20 ^{vi}	2.9809	K20—O15 ^{xx}	2.8262
O12—H21	1.0546	K20—O16 ^{xv}	2.8476
O13—C5	1.2700	K20—O17 ⁱ	2.9139
O13—K19 ^{vii}	2.7324	H21—O12	1.0546
O13—K20 ⁱⁱⁱ	2.9018	H21—O15 ⁱ	1.4301
C2—C1—O11	121.205	H9 ⁱⁱⁱ —C4—H10 ⁱⁱⁱ	108.742
C2—C1—O12	118.533	C4—C5—O13	118.164
O11—C1—O12	120.209	C4—C5—O14	117.295
C1—C2—C3	118.102	O13—C5—O14	124.523
C1—C2—H7	107.728	C3—C6—O15 ⁱ	116.534
C3—C2—H7	108.894	C3—C6—O16 ^{iv}	119.29
C1—C2—H8	106.997	O15 ⁱ —C6—O16 ^{iv}	124.176
C3—C2—H8	107.477	C1—O11—K19	160.328
H7—C2—H8	107.155	C1—O12—H21	112.363
C2—C3—C4 ⁱ	110.361	C5—O13—K19 ^{vii}	151.738
C2—C3—C6	111.212	C5—O14—K20 ^{ix}	136.399
C4 ⁱ —C3—C6	110.672	C5—O14—K20 ^x	118.025
C2—C3—O17	104.643	K20 ^{ix} —O14—K20 ^x	93.08
C4 ⁱ —C3—O17	109.444	C6 ^{xii} —O16—K19 ^{xiii}	155.314
C6—C3—O17	110.341	C3—O17—H18	112.048
C3 ⁱⁱ —C4—C5	111.776	O11—K19—O13 ^{xvi}	75.177
C3 ⁱⁱ —C4—H9 ⁱⁱⁱ	108.013	O11—K19—O16 ^{xviii}	99.027
C5—C4—H9 ⁱⁱⁱ	110.083	O13 ^{xvi} —K19—O16 ^{xviii}	68.431
C3 ⁱⁱ —C4—H10 ⁱⁱⁱ	109.581	O14 ^{xx} —K20—O14 ^x	86.92
C5—C4—H10 ⁱⁱⁱ	108.6		

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

(ZZZQRO01_DFT_2020)

Crystal data

2K⁺·C₆H₆O₇²⁻ c = 18.9180 Å
M_r = 268.3 β = 90.0900°
 Monoclinic, C2/C V = 1963.78 Å³
a = 10.7570 Å Z = 8
b = 9.6500 Å

Data collection

DFT calculation k = →
h = → l = →

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.25901	0.04541	0.22942	0.03000*
C2	0.18032	0.06770	0.29481	0.03000*
C3	0.24734	0.09830	0.36529	0.03000*
C4	0.84937	0.70884	0.35433	0.03000*
C5	0.90431	0.75739	0.42438	0.03000*
C6	0.30314	-0.03537	0.39758	0.03000*
H7	0.12145	-0.02382	0.30156	0.03900*
H8	0.11908	0.15470	0.28379	0.03900*
H9	-0.19189	-0.20405	0.32631	0.03900*
H10	-0.07615	-0.33414	0.32205	0.03900*
O11	0.23110	0.09838	0.17278	0.03000*
O12	0.35671	-0.03752	0.23393	0.03000*
O13	0.87503	0.87696	0.44664	0.03000*
O14	0.97406	0.67389	0.45700	0.03000*
O15	0.88346	0.40040	0.36001	0.03000*
O16	0.26835	0.92611	0.45712	0.03000*
O17	0.15267	0.15276	0.41038	0.03000*
H18	0.16110	0.11919	0.45951	0.03900*
K19	0.10153	0.15846	0.05435	0.03000*
K20	-0.38269	-0.04931	0.42269	0.03000*
H21	0.37493	-0.06869	0.28630	0.03900*

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>
O12—H21···O15	1.053	1.429	2.476	171.5
O17—H18···O13	0.988	1.819	2.737	153.1

(acig037_19_DFT)

Crystal data

K⁺·Rb⁺·C₆H₆O₇²⁻ a = 5.3536 Å
M_r = 314.6 b = 16.2869 Å
 Monoclinic, P2₁/C c = 11.1813 Å

$\beta = 91.0480^\circ$
 $V = 974.77 \text{ \AA}^3$

$Z = 4$

Data collection

DFT 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.348600	0.596178	0.237535	0.03600*
C2	0.238715	0.527039	0.162864	0.34800*
C3	0.243120	0.440213	0.221644	0.34800*
C4	0.186510	0.377530	0.121968	0.34800*
C5	0.233878	0.286924	0.147671	0.03600*
C6	0.501044	0.423154	0.275443	0.03600*
H7	0.042956	0.541776	0.144154	0.45300*
H8	0.337221	0.525072	0.078723	0.45300*
H9	0.299340	0.391976	0.044813	0.45300*
H10	-0.007836	0.384081	0.093125	0.45300*
O11	0.246780	0.625594	0.325481	0.03600*
O12	0.562683	0.624261	0.195748	0.03600*
O13	0.230775	0.240197	0.058309	0.03600*
O14	0.273553	0.262297	0.255030	0.03600*
O15	0.662962	0.395699	0.204575	0.03600*
O16	0.542487	0.438473	0.384327	0.03600*
O17	0.067740	0.437431	0.315916	0.03600*
H18	-0.094627	0.420386	0.278037	0.04700*
K19	0.802278	0.925707	0.941692	0.03410*
Rb20	0.260970	0.728191	0.563240	0.03410*
H21	0.634323	0.682625	0.229966	0.05000*

Bond lengths (\AA)

C1—C2	1.501	C4—C5	1.516
C1—O11	1.236	C4—H9	1.092
C1—O12	1.321	C4—H10	1.087
C2—C3	1.561	C5—O13	1.255
C2—H7	1.091	C5—O14	1.281
C2—H8	1.086	C6—O15	1.264
C3—C4	1.535	C6—O16	1.261
C3—C6	1.520	O12—H21	1.073
C3—O17	1.422	O17—H18	0.989

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O12—H21 \cdots O14	1.092	1.397	2.471	166.4
O17—H18 \cdots O15	0.999	1.575	2.571	173.6

(acig037_20_DFT)

Crystal data $\text{K}^+\cdot\text{Rb}^+\cdot\text{C}_6\text{H}_6\text{O}_7^{2-}$ $M_r = 314.6$ Monoclinic, $P2_1/C$ $a = 5.3536 \text{ \AA}$ $b = 16.2869 \text{ \AA}$ $c = 11.1813 \text{ \AA}$ $\beta = 91.0480^\circ$ $V = 974.77 \text{ \AA}^3$ $Z = 4$ *Data collection*

DFT 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.30032	0.59865	0.25001	0.03600*
C2	0.21398	0.53481	0.16164	0.34800*
C3	0.26317	0.44413	0.20081	0.34800*
C4	0.22517	0.38992	0.08961	0.34800*
C5	0.29104	0.29994	0.10461	0.03600*
C6	0.53062	0.43948	0.24835	0.03600*
H7	0.01271	0.54199	0.14887	0.45300*
H8	0.30266	0.54614	0.07664	0.45300*
H9	0.33899	0.41314	0.01697	0.45300*
H10	0.03081	0.39446	0.06041	0.45300*
O11	0.26107	0.59516	0.35850	0.03600*
O12	0.42390	0.66083	0.20363	0.03600*
O13	0.17053	0.24799	0.04403	0.03600*
O14	0.47940	0.28164	0.17171	0.03600*
O15	0.69376	0.46303	0.17563	0.03600*
O16	0.57619	0.41930	0.35541	0.03600*
O17	0.09972	0.41890	0.29262	0.03600*
H18	-0.06980	0.42924	0.25924	0.04700*
Rb19	0.77465	0.92825	0.95427	0.03410*
K20	0.28135	0.71996	0.54559	0.03410*
H21	0.47197	0.70794	0.26709	0.05000*

Bond lengths (\AA)

C1—C2	1.501	C4—C5	1.516
C1—O11	1.236	C4—H9	1.092
C1—O12	1.321	C4—H10	1.087
C2—C3	1.561	C5—O13	1.255
C2—H7	1.091	C5—O14	1.281
C2—H8	1.086	C6—O15	1.264
C3—C4	1.535	C6—O16	1.261
C3—C6	1.520	O12—H21	1.073
C3—O17	1.422	O17—H18	0.989

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O12—H21 \cdots O14	1.073	1.404	2.461	166.8
O17—H18 \cdots O15	0.989	1.654	2.617	163.2

Potassium rubidium hydrogen citrate (kadu1880_phase_1)

Crystal data

0.286K⁺·1.714Rb⁺·C₆H₆O₇²⁻

M_r = 347.79

Monoclinic, *P*2₁

a = 5.9507 (3) Å

b = 14.9519 (5) Å

c = 5.3342 (3) Å

β = 94.5623 (18)°

V = 473.11 (5) Å³

Z = 2

D_x = 2.441 Mg m⁻³

T = 300 K

flat_sheet, 25 × 25 mm

Data collection

Bruker D2 Phaser
diffractometer

Specimen mounting: PMMA holder

Data collection mode: reflection

Scan method: step

Refinement

Profile function: Crystallite size in microns with
"isotropic" model: parameters: Size, G/L mix
1.000, 1.000, Microstrain, "isotropic" model
(10⁶ * delta Q/Q) parameters: Mustrain, G/L
mix 3.35(5)e3, 1.000,

0 restraints

Preferred orientation correction: March-Dollase
correction coef. = 1.000 axis = [0, 0, 1]

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}	Occ. (<1)
C1	0.76300	0.79690	0.52690	0.042*	
C2	0.89060	0.71880	0.43250	0.012*	
C3	0.78580	0.63310	0.52740	0.012*	
C4	0.88190	0.55210	0.39600	0.012*	
C5	0.77880	0.46920	0.49700	0.042*	
C6	0.53530	0.63560	0.46770	0.042*	
O7	0.58700	0.82360	0.40100	0.042*	
O8	0.82000	0.82960	0.74210	0.042*	
O9	0.40330	0.63500	0.64610	0.042*	
O10	0.45740	0.63400	0.23890	0.042*	
O11	0.87650	0.43030	0.68590	0.042*	
O12	0.62250	0.43070	0.36400	0.042*	
O13	0.84380	0.62580	0.79950	0.042*	
H14	1.06360	0.72270	0.47410	0.015*	
H15	0.85100	0.71660	0.20170	0.015*	
H16	0.69000	0.64450	0.89910	0.054*	
H17	1.07640	0.54810	0.45930	0.015*	

H18	0.86160	0.55470	0.18930	0.015*	
Rb19	0.27620	0.49035	-0.02780	0.064*	0.87
Rb20	0.73100	0.27937	0.03490	0.064*	0.84
H21	0.51460	0.38030	0.48000	0.054*	
K22	0.27620	0.49035	-0.02780	0.064*	0.13
K23	0.73100	0.27937	0.03490	0.064*	0.16

Geometric parameters (Å, °)

C1—C2	1.5015	H17—C4	1.1811
C1—O7	1.2631	H17—Rb19 ^{vii}	3.0662
C1—O8	1.2688	H17—Rb19 ^v	3.0211
C2—C1	1.5015	H18—C4	1.1003
C2—C3	1.5288	H18—Rb19 ^{vii}	2.9679
C2—H14	1.0374	Rb19—O8 ^{viii}	2.9268
C2—H15	1.2354	Rb19—O9 ^{ix}	2.9129
C3—C2	1.5288	Rb19—O10	2.7488
C3—C4	1.5326	Rb19—O11 ^x	2.8667
C3—C6	1.4998	Rb19—O12	2.9538
C3—O13	1.4690	Rb19—H17 ^x	3.0211
C4—C3	1.5326	Rb19—H17 ^{xi}	3.0662
C4—C5	1.5019	Rb19—H18 ^{xi}	2.9679
C4—H17	1.1811	Rb19—Rb20 ^{xi}	4.5565
C4—H18	1.1003	Rb19—Rb20	4.1526
C5—C4	1.5019	Rb19—Rb20 ⁱ	4.3218
C5—O11	1.2643	Rb20—O7 ^{xii}	2.9538
C5—O12	1.2629	Rb20—O8 ^{xiii}	2.9347
C6—C3	1.4998	Rb20—O9 ^{viii}	2.9000
C6—O9	1.2812	Rb20—O10 ^{xii}	2.8025
C6—O10	1.2708	Rb20—O11 ^{ix}	3.0926
O7—C1	1.2631	Rb20—O12	2.9662
O7—Rb20 ⁱ	2.9538	Rb20—H14 ^{xiii}	2.9261
O7—H21 ⁱⁱ	1.2443	Rb20—H15 ^{xiv}	3.0266
O8—C1	1.2688	Rb20—Rb19	4.1526
O8—Rb19 ⁱⁱ	2.9268	Rb20—Rb19 ^{vii}	4.5565
O8—Rb20 ⁱⁱⁱ	2.9347	Rb20—Rb19 ^{xii}	4.3218
O9—C6	1.2812	H21—O7 ^{viii}	1.2443
O9—Rb19 ^{iv}	2.9129	H21—O12	1.1943
O9—Rb20 ⁱⁱ	2.9000	K22—O8 ^{viii}	2.9268
O10—C6	1.2708	K22—O9 ^{ix}	2.9129
O10—Rb19	2.7488	K22—O10	2.7488
O10—Rb20 ⁱ	2.8025	K22—O11 ^x	2.8667
O11—C5	1.2643	K22—O12	2.9538
O11—Rb19 ^v	2.8667	K22—H17 ^x	3.0211
O11—Rb20 ^{iv}	3.0926	K22—H18 ^{xi}	2.9679
O12—C5	1.2629	K22—Rb20	4.1526
O12—Rb19	2.9538	K22—Rb20 ⁱ	4.3218
O12—Rb20	2.9662	K23—O7 ^{xii}	2.9538

O12—H21	1.1943	K23—O8 ^{xiii}	2.9347
O13—C3	1.4690	K23—O9 ^{viii}	2.9000
O13—H16	1.1294	K23—O10 ^{xii}	2.8025
H14—C2	1.0374	K23—O11 ^{ix}	3.0926
H14—Rb20 ⁱⁱⁱ	2.9261	K23—O12	2.9662
H15—C2	1.2354	K23—H14 ^{xiii}	2.9261
H15—Rb20 ^{vi}	3.0266	K23—Rb19	4.1526
H16—O13	1.1294	K23—Rb19 ^{xii}	4.3218
C2—C1—O7	119.179	C6—O9—Rb19 ^{iv}	130.722
C2—C1—O8	119.812	C6—O9—Rb20 ⁱⁱ	130.123
O7—C1—O8	120.659	Rb19 ^{iv} —O9—Rb20 ⁱⁱ	96.057
C1—C2—C3	108.078	C6—O10—Rb19	127.579
C1—C2—H14	113.845	C6—O10—Rb20 ⁱ	125.884
C3—C2—H14	113.597	Rb19—O10—Rb20 ⁱ	102.246
C1—C2—H15	107.065	C5—O11—Rb19 ^v	126.154
C3—C2—H15	104.902	C5—O12—H21	113.508
H14—C2—H15	108.788	C3—O13—H16	108.114
C2—C3—C4	109.513	O9 ^{ix} —Rb19—O10	67.61
C2—C3—C6	109.483	O9 ^{ix} —Rb19—O11 ^x	99.106
C4—C3—C6	108.863	O10—Rb19—O11 ^x	142.669
C2—C3—O13	108.63	O9 ^{ix} —Rb19—Rb20	114.433
C4—C3—O13	109.042	O10—Rb19—Rb20	109.001
C6—C3—O13	111.29	O11 ^x —Rb19—Rb20	108.228
C3—C4—C5	108.071	O9 ^{ix} —Rb19—Rb20 ⁱ	41.858
C3—C4—H17	107.583	O10—Rb19—Rb20 ⁱ	39.324
C5—C4—H17	105.94	O11 ^x —Rb19—Rb20 ⁱ	107.535
C3—C4—H18	114.529	Rb20—Rb19—Rb20 ⁱ	140.019
C5—C4—H18	111.914	O9 ^{viii} —Rb20—O10 ^{xii}	67.107
H17—C4—H18	108.376	O9 ^{viii} —Rb20—Rb19	113.857
C4—C5—O11	119.374	O10 ^{xii} —Rb20—Rb19	108.425
C4—C5—O12	118.575	O9 ^{viii} —Rb20—Rb19 ^{xii}	42.086
O11—C5—O12	120.629	O10 ^{xii} —Rb20—Rb19 ^{xii}	38.43
C3—C6—O9	119.989	Rb19—Rb20—Rb19 ^{xii}	138.847
C3—C6—O10	118.941	O7 ^{viii} —H21—O12	175.812
O9—C6—O10	120.985	O10—K22—Rb20	109.001
C1—O7—H21 ⁱⁱ	111.106		

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

Potassium rubidium hydrogen citrate (kadu1880_phase_0)

Crystal data

$1.668\text{K}^+ \cdot 0.332\text{Rb}^+ \cdot \text{C}_6\text{H}_6\text{O}_7^{2-}$

$M_r = 283.68$

Monoclinic, $C2/c$

$a = 10.8409 (7) \text{ \AA}$

$b = 9.6926 (6) \text{ \AA}$

$c = 18.9810 (13) \text{ \AA}$

$\beta = 90.150 (6)^\circ$

$V = 1994.4 (2) \text{ \AA}^3$

$Z = 8$

$D_x = 1.890 \text{ Mg m}^{-3}$

$T = 300 \text{ K}$

flat_sheet, $25 \times 25 \text{ mm}$

Data collection

Bruker D2 Phaser
diffractometer
Specimen mounting: PMMA holder

Data collection mode: reflection
Scan method: step

Refinement

Profile function: Crystallite size in microns with
"isotropic" model: parameters: Size, G/L mix
1.000, 1.000, Microstrain, "isotropic" model
(10⁶ * delta Q/Q) parameters: Mustrain, G/L
mix 2.86(12)e3, 1.000,
0 restraints
Preferred orientation correction: March-Dollase
correction coef. = 1.000 axis = [0, 0, 1]

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}	Occ. (<1)
C1	0.25901	0.04541	0.22942	0.030*	
C2	0.18032	0.06770	0.29481	0.030*	
C3	0.24734	0.09830	0.36529	0.030*	
C4	0.84937	0.70884	0.35433	0.030*	
C5	0.90431	0.75739	0.42438	0.030*	
C6	0.30314	-0.03537	0.39758	0.030*	
H7	0.12145	-0.02382	0.30156	0.039*	
H8	0.11908	0.15470	0.28379	0.039*	
H9	-0.19189	-0.20405	0.32631	0.039*	
H10	-0.07615	-0.33414	0.32205	0.039*	
O11	0.23110	0.09838	0.17278	0.030*	
O12	0.35671	-0.03752	0.23393	0.030*	
O13	0.87503	0.87696	0.44664	0.030*	
O14	0.97406	0.67389	0.45700	0.030*	
O15	0.88346	0.40040	0.36001	0.030*	
O16	0.26835	0.92611	0.45712	0.030*	
O17	0.15267	0.15276	0.41038	0.030*	
H18	0.16110	0.11919	0.45951	0.039*	
K19	0.10153	0.15846	0.05435	0.030*	0.799
Rb20	0.10153	0.15846	0.05435	0.030*	0.201
K21	-0.38269	-0.04931	0.42269	0.030*	0.870
Rb22	-0.38269	-0.04931	0.42269	0.030*	0.130
H23	0.37493	-0.06869	0.28630	0.039*	

Geometric parameters (Å, °)

C1—C2	1.5233	H18—K19 ^{vii}	2.8838
C1—O11	1.2285	K19—O11	2.7109
C1—O12	1.3322	K19—O13 ^{xvii}	2.7403
C2—C1	1.5233	K19—O14 ^{xviii}	2.8198
C2—C3	1.5493	K19—O15 ^{xii}	2.8577
C2—H7	1.1005	K19—O16 ^{xix}	2.7140

C2—H8	1.0931	K19—O16 ^v	2.9612
C3—C2	1.5493	K19—O17 ^{vii}	2.8381
C3—C4 ⁱ	1.5543	K19—H18 ^{vii}	2.8838
C3—C6	1.5551	K19—K19 ^{xx}	4.3029
C3—O17	1.4384	K19—K19 ^{xxi}	4.2194
C4—C3 ⁱⁱ	1.5543	K19—K21 ^{vii}	3.6782
C4—C5	1.5297	K19—K21 ^{xxii}	3.7211
C4—H9 ⁱⁱⁱ	1.0930	Rb20—C6 ^{xv}	3.2714
C4—H10 ⁱⁱⁱ	1.0970	Rb20—O11	2.7109
C5—C4	1.5297	Rb20—O13 ^{xvii}	2.7403
C5—O13	1.2740	Rb20—O13 ^{xviii}	3.2095
C5—O14	1.2679	Rb20—O14 ^{xviii}	2.8198
C6—C3	1.5551	Rb20—O15 ^{xii}	2.8577
C6—O15 ⁱ	1.2875	Rb20—O16 ^{xix}	2.7140
C6—O16 ^{iv}	1.2494	Rb20—O16 ^v	2.9612
C6—Rb20 ^v	3.2714	Rb20—O17 ^{vii}	2.8381
H7—C2	1.1005	Rb20—H18 ^{vii}	2.8838
H8—C2	1.0931	Rb20—K19 ^{xx}	4.3029
H9—C4 ^{vi}	1.0930	Rb20—K19 ^{xxi}	4.2194
H9—Rb22	3.1455	Rb20—K21 ^{vii}	3.6782
H10—C4 ^{vi}	1.0970	Rb20—K21 ^{xxii}	3.7211
O11—C1	1.2285	Rb20—Rb22 ^{xxiii}	4.5418
O11—K19	2.7109	K21—O11 ^{vii}	2.8371
O11—K21 ^{vii}	2.8371	K21—O12 ^{vii}	2.9890
O12—C1	1.3322	K21—O13 ^{vi}	2.9183
O12—K21 ^{vii}	2.9890	K21—O14 ^{xxiv}	2.7425
O12—H23	1.0571	K21—O14 ^{xi}	2.7687
O13—C5	1.2740	K21—O15 ^{xxiv}	2.8398
O13—K19 ^{viii}	2.7403	K21—O16 ^{xvi}	2.8552
O13—Rb20 ^{ix}	3.2095	K21—O17 ⁱ	2.9225
O13—K21 ⁱⁱⁱ	2.9183	K21—K19 ^{vii}	3.6782
O14—C5	1.2679	K21—K19 ^{xxv}	3.7211
O14—K19 ^{ix}	2.8198	K21—K21 ^{xxvi}	4.0044
O14—K21 ^x	2.7425	Rb22—H9	3.1455
O14—K21 ^{xi}	2.7687	Rb22—O11 ^{vii}	2.8371
O15—C6 ⁱⁱ	1.2875	Rb22—O12 ^{vii}	2.9890
O15—K19 ^{xii}	2.8577	Rb22—O13 ^{vi}	2.9183
O15—K21 ^x	2.8398	Rb22—O14 ^{xxiv}	2.7425
O15—H23 ⁱⁱ	1.4336	Rb22—O14 ^{xi}	2.7687
O16—C6 ^{xiii}	1.2494	Rb22—O15 ^{xxiv}	2.8398
O16—K19 ^{xiv}	2.7140	Rb22—O16 ^{xvi}	2.8552
O16—K19 ^{xv}	2.9612	Rb22—O17 ⁱ	2.9225
O16—K21 ^{xvi}	2.8552	Rb22—K19 ^{vii}	3.6782
O17—C3	1.4384	Rb22—K19 ^{xxv}	3.7211
O17—H18	0.9917	Rb22—Rb20 ^{xxvii}	4.5418
O17—K19 ^{vii}	2.8381	Rb22—K21 ^{xxvi}	4.0044
O17—K21 ⁱⁱ	2.9225	H23—O12	1.0571
H18—O17	0.9917	H23—O15 ⁱ	1.4336

C2—C1—O11	121.104	O16 ^{xix} —Rb20—K21 ^{vii}	50.339
C2—C1—O12	118.689	O17 ^{vii} —Rb20—K21 ^{vii}	140.016
O11—C1—O12	120.155	O11—Rb20—K21 ^{xxii}	113.299
C1—C2—C3	117.926	O13 ^{xvii} —Rb20—K21 ^{xxii}	144.871
C1—C2—H7	107.861	O14 ^{xviii} —Rb20—K21 ^{xxii}	47.669
C3—C2—H7	108.94	O15 ^{xii} —Rb20—K21 ^{xxii}	49.014
C1—C2—H8	107.136	O16 ^{xix} —Rb20—K21 ^{xxii}	137.367
C3—C2—H8	107.503	O17 ^{vii} —Rb20—K21 ^{xxii}	50.762
H7—C2—H8	106.978	K21 ^{vii} —Rb20—K21 ^{xxii}	158.634
C2—C3—C4 ⁱ	110.406	O14 ^{xxiv} —K21—O14 ^{xi}	86.798
C2—C3—C6	111.216	O14 ^{xxiv} —K21—K19 ^{vii}	90.547
C4 ⁱ —C3—C6	110.538	O14 ^{xi} —K21—K19 ^{vii}	116.053
C2—C3—O17	104.495	O14 ^{xxiv} —K21—K19 ^{xxv}	102.184
C4 ⁱ —C3—O17	109.62	O14 ^{xi} —K21—K19 ^{xxv}	48.842
C6—C3—O17	110.405	K19 ^{vii} —K21—K19 ^{xxv}	158.634
C3 ⁱⁱ —C4—C5	111.797	O14 ^{xxiv} —K21—K21 ^{xxvi}	43.657
C3 ⁱⁱ —C4—H9 ⁱⁱⁱ	107.888	O14 ^{xi} —K21—K21 ^{xxvi}	43.141
C5—C4—H9 ⁱⁱⁱ	110.074	K19 ^{vii} —K21—K21 ^{xxvi}	108.072
C3 ⁱⁱ —C4—H10 ⁱⁱⁱ	109.758	K19 ^{xxv} —K21—K21 ^{xxvi}	71.91
C5—C4—H10 ⁱⁱⁱ	108.471	O11 ^{vii} —Rb22—O13 ^{vi}	70.558
H9 ⁱⁱⁱ —C4—H10 ⁱⁱⁱ	108.811	O11 ^{vii} —Rb22—O14 ^{xxiv}	94.759
C4—C5—O13	118.121	O13 ^{vi} —Rb22—O14 ^{xxiv}	134.319
C4—C5—O14	117.286	O11 ^{vii} —Rb22—O14 ^{xi}	162.926
O13—C5—O14	124.575	O13 ^{vi} —Rb22—O14 ^{xi}	96.266
C3—C6—O15 ⁱ	116.585	O14 ^{xxiv} —Rb22—O14 ^{xi}	86.798
C3—C6—O16 ^{iv}	119.172	O11 ^{vii} —Rb22—O15 ^{xxiv}	109.693
O15 ⁱ —C6—O16 ^{iv}	124.243	O13 ^{vi} —Rb22—O15 ^{xxiv}	151.221
C1—O11—K19	160.277	O14 ^{xxiv} —Rb22—O15 ^{xxiv}	74.302
C1—O12—H23	112.299	O14 ^{xi} —Rb22—O15 ^{xxiv}	87.121
C5—O13—K19 ^{viii}	151.675	O11 ^{vii} —Rb22—O16 ^{xvi}	92.77
C5—O14—K21 ^x	136.46	O13 ^{vi} —Rb22—O16 ^{xvi}	64.183
C5—O14—K21 ^{xi}	117.837	O14 ^{xxiv} —Rb22—O16 ^{xvi}	74.054
K21 ^x —O14—K21 ^{xi}	93.202	O14 ^{xi} —Rb22—O16 ^{xvi}	71.27
C6 ^{xiii} —O16—K19 ^{xiv}	155.31	O15 ^{xxiv} —Rb22—O16 ^{xvi}	142.351
C3—O17—H18	112.023	O11 ^{vii} —Rb22—O17 ⁱ	111.788
O11—K19—O13 ^{xvii}	75.164	O13 ^{vi} —Rb22—O17 ⁱ	69.192
O11—K19—O16 ^{xix}	98.873	O14 ^{xxiv} —Rb22—O17 ⁱ	150.799
O13 ^{xvii} —K19—O16 ^{xix}	68.448	O14 ^{xi} —Rb22—O17 ⁱ	71.476
O11—K19—K21 ^{vii}	49.962	O15 ^{xxiv} —Rb22—O17 ⁱ	85.069
O13 ^{xvii} —K19—K21 ^{vii}	51.6	O16 ^{xvi} —Rb22—O17 ⁱ	114.858
O16 ^{xix} —K19—K21 ^{vii}	50.339	O11 ^{vii} —Rb22—K19 ^{vii}	47.017
O11—K19—K21 ^{xxii}	113.299	O13 ^{vi} —Rb22—K19 ^{vii}	47.383
O13 ^{xvii} —K19—K21 ^{xxii}	144.871	O14 ^{xxiv} —Rb22—K19 ^{vii}	90.547
O16 ^{xix} —K19—K21 ^{xxii}	137.367	O14 ^{xi} —Rb22—K19 ^{vii}	116.053
K21 ^{vii} —K19—K21 ^{xxii}	158.634	O15 ^{xxiv} —Rb22—K19 ^{vii}	151.912
O11—Rb20—O13 ^{xvii}	75.164	O16 ^{xvi} —Rb22—K19 ^{vii}	47.035
O11—Rb20—O14 ^{xviii}	156.805	O17 ⁱ —Rb22—K19 ^{vii}	116.209

O13 ^{xvii} —Rb20—O14 ^{xviii}	127.921	O11 ^{vii} —Rb22—K19 ^{xxv}	146.039
O11—Rb20—O15 ^{xii}	71.094	O13 ^{vi} —Rb22—K19 ^{xxv}	113.939
O13 ^{xvii} —Rb20—O15 ^{xii}	144.666	O14 ^{xxiv} —Rb22—K19 ^{xxv}	102.184
O14 ^{xviii} —Rb20—O15 ^{xii}	85.81	O14 ^{xi} —Rb22—K19 ^{xxv}	48.842
O11—Rb20—O16 ^{xix}	98.873	O15 ^{xxiv} —Rb22—K19 ^{xxv}	49.433
O13 ^{xvii} —Rb20—O16 ^{xix}	68.448	O16 ^{xvi} —Rb22—K19 ^{xxv}	120.024
O14 ^{xviii} —Rb20—O16 ^{xix}	93.138	O17 ⁱ —Rb22—K19 ^{xxv}	48.779
O15 ^{xii} —Rb20—O16 ^{xix}	126.697	K19 ^{vii} —Rb22—K19 ^{xxv}	158.634
O11—Rb20—O17 ^{vii}	107.551	O11 ^{vii} —Rb22—K21 ^{xxvi}	135.87
O13 ^{xvii} —Rb20—O17 ^{vii}	94.149	O13 ^{vi} —Rb22—K21 ^{xxvi}	123.639
O14 ^{xviii} —Rb20—O17 ^{vii}	72.02	O14 ^{xxiv} —Rb22—K21 ^{xxvi}	43.657
O15 ^{xii} —Rb20—O17 ^{vii}	86.312	O14 ^{xi} —Rb22—K21 ^{xxvi}	43.141
O16 ^{xix} —Rb20—O17 ^{vii}	143.481	O15 ^{xxiv} —Rb22—K21 ^{xxvi}	77.289
O11—Rb20—K21 ^{vii}	49.962	O16 ^{xvi} —Rb22—K21 ^{xxvi}	65.784
O13 ^{xvii} —Rb20—K21 ^{vii}	51.6	O17 ⁱ —Rb22—K21 ^{xxvi}	112.22
O14 ^{xviii} —Rb20—K21 ^{vii}	142.911	K19 ^{vii} —Rb22—K21 ^{xxvi}	108.072
O15 ^{xii} —Rb20—K21 ^{vii}	109.63	K19 ^{xxv} —Rb22—K21 ^{xxvi}	71.91

Symmetry codes: (i) $x-1/2, y-1/2, z$; (ii) $x+1/2, y+1/2, z$; (iii) $x+1, y+1, z$; (iv) $x, y-1, z$; (v) $-x+1/2, y-1/2, -z+1/2$; (vi) $x-1, y-1, z$; (vii) $-x, y, -z+1/2$; (viii) $-x+1, y+1, -z+1/2$; (ix) $x+1, -y+1, z+3/2$; (x) $x+3/2, y+1/2, z$; (xi) $-x+1/2, -y+1/2, -z+1$; (xii) $-x+1, y, -z+1/2$; (xiii) $x, y+1, z$; (xiv) $x, -y+1, z+3/2$; (xv) $-x+1/2, y+1/2, -z+1/2$; (xvi) $-x, -y+1, -z+1$; (xvii) $-x+1, y-1, -z+1/2$; (xviii) $x-1, -y+1, z+1/2$; (xix) $x, -y+1, z+1/2$; (xx) $-x, -y, -z$; (xxi) $-x+1/2, -y+1/2, -z$; (xxii) $-x-1/2, y+1/2, -z+1/2$; (xxiii) $x+1/2, -y+1/2, z+1/2$; (xxiv) $x-3/2, y-1/2, z$; (xxv) $-x-1/2, y-1/2, -z+1/2$; (xxvi) $-x-1, -y, -z+1$; (xxvii) $x-1/2, -y+1/2, z+3/2$.