Crystal structure of anhydrous tripotassium citrate from laboratory X-ray powder diffraction data and DFT comparison

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The crystal structure of anhydrous tripotassium citrate, $[K_3(C_6H_5O_7)]_n$, has been solved and refined using laboratory X-ray powder diffraction data, and optimized using density functional techniques. The three unique potassium cations are 6-, 8-, and 6-coordinate (all irregular). The $[KO_n]$ coordination polyhedra share edges and corners to form a three-dimensional framework, with channels running parallel to the c axis. The only hydrogen bond is an intramolecular one involving the hydroxy group and the central carboxylate group, with graph-set motif $S(5)$.

1. Chemical context

In the course of a systematic study of the crystal structures of group 1 (alkali metal) citrate salts to understand the anion's conformational flexibility, ionization, coordination tendencies, and hydrogen bonding, we have determined several new crystal structures. Most of the new structures were solved using powder X-ray 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, 2016*a*). Five of the new structures, *viz.* $NaKHC_6H_5O_7$, $NaK_2C_6H_5O_7$, $Na_3C_6H_5O_7$, $NaH_2C_6H_5O_7$, and $Na_2HC_6H_5O_7$, have been published recently (Rammohan & Kaduk, 2016*b,c,d,e*; Rammohan *et al.*, 2016), and two additional structures, *viz.* $KH_2C_6H_5O_7$ and $KH_2C_6H_5O_7(H_2O)_2$, have been communicated to the Cambridge Structural Database (Kaduk & Stern, 2016*a,b*).

http://dx.doi.org/10.1107/S2056989016011506 **1159**
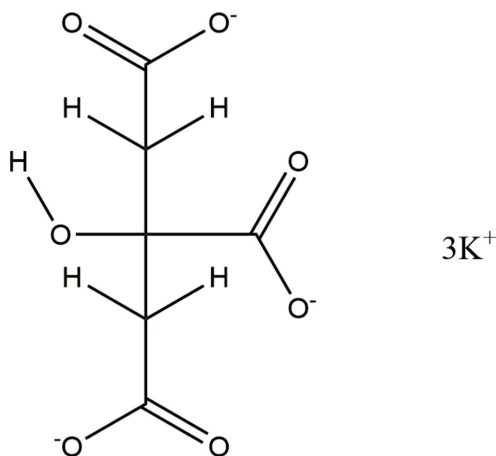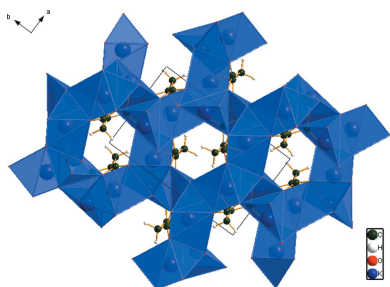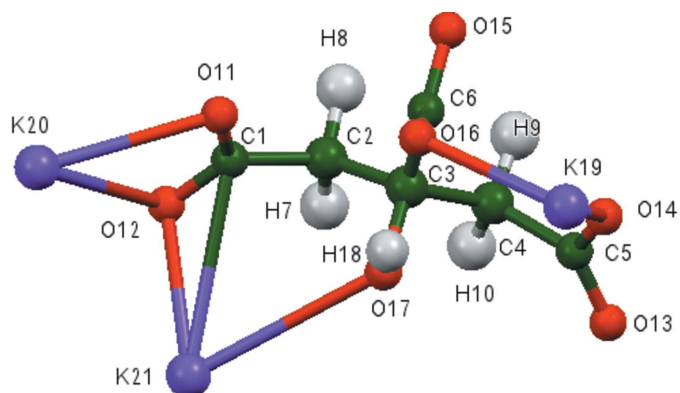
Figure 1
The asymmetric unit of the title compound, showing the atom numbering. Atoms are represented by 50% probability spheroids.

2. Structural commentary

The asymmetric unit of the title compound is shown in Fig. 1. The r.m.s. deviation of the non-hydrogen atoms between the Rietveld-refined and the DFT-optimized structures is 0.117 Å (Fig. 2). The maximum deviation is 0.260 Å, at O14. The good agreement between the two structures is strong evidence that the experimental structure is correct (van de Streek & Neumann, 2014). This discussion uses the DFT-optimized structure. Most of the bond lengths, bond angles, and torsion angles fall within the normal ranges indicated by a *Mercury Mogul* Geometry Check (Macrae *et al.*, 2008). Only the C4—C5 bond length [refined = 1.511 (5), optimized = 1.536, *Mogul* average = 1.498 (12) Å, Z-score = 3.1], and the C3—C2—C1 [refined = 115 (2), optimized = 115.0, *Mogul* average = 103 (2)°] and O17—C3—C2 angles [refined = 107 (2), optimized = 109.6, Mogul average = 106 (2)°] are flagged as unusual. 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 occur in the normal planar arrangement. Both
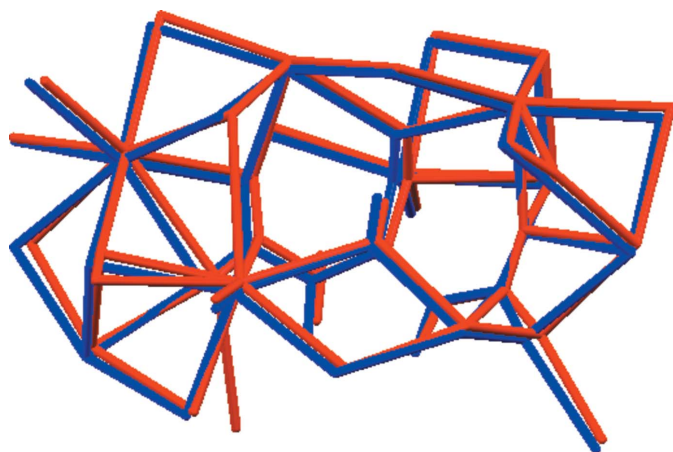
Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O17—H18$\cdots$O16	0.983	1.814	2.552	129.1

terminal carboxylate groups O11/O12 and O13/O14 chelate to a single potassium cation (K20 for each). The terminal carboxylate oxygen atom O12 and the hydroxy O17 atom chelate to K21, and the terminal carboxylate oxygen atoms O13 and O17 chelate to K19. The terminal/central pairs O11/O16, O14/O16, O11/O15, and O14/O15 chelate to K21, K19, K19, and K21, respectively. The three potassium cations K19, K20, and K21 are 6-, 8-, and 6-coordinate, respectively (all irregular, using a K—O cut-off distance of 3.24 Å). Their bond-valence sums are 1.12, 1.03, and 1.12 valence units. The metal-oxygen bonding is ionic, based on the cation charges and the Mulliken overlap populations.

Although the lattice parameters of anhydrous tripotassium citrate are in general similar to those of the monohydrate (Carrell *et al.*, 1987; CSD code ZZZHVI01), consistent with the difference in water content, the powder patterns differ considerably. Visual examination of the structures shows that the arrangements of the citrate anions are very different. A mechanism for the transformation of one phase into the other is not obvious.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) morphology suggests that we might expect blocky morphology for anhydrous tripotassium citrate, with {011} as the principal faces. A second-order spherical harmonic texture model was included in the refinement. The texture index was only 1.001, indicating that preferred orientation was not significant for this rotated flat plate specimen.

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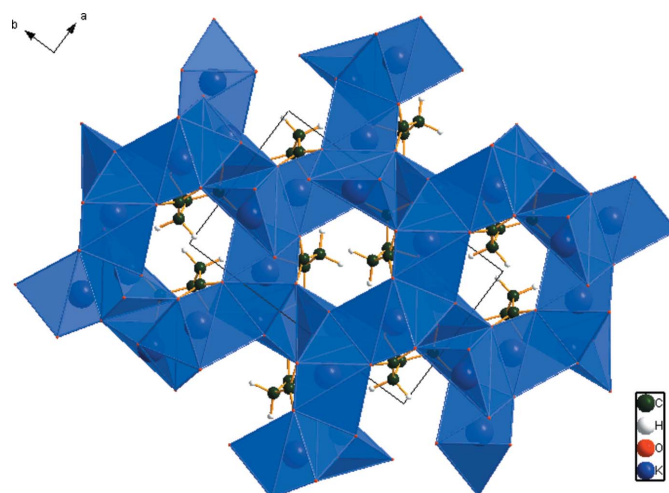

Figure 3
The crystal structure of $K_3C_6H_5O_7$, viewed down the *c* axis, with coordination spheres of the potassium cations in polyhedral representation.

Table 2
Experimental details.

	Phase 1	Phase 2
Crystal data		
Chemical formula	$[K_3(C_6H_5O_7)]$	Si
M_r	306.39	28.09
Crystal system, space group	Orthorhombic, $Pna2_1$	Cubic, $Fd\bar{3}m$
Temperature (K)	300	300
a, b, c (Å)	7.7062 (2), 12.4693 (3), 10.4241 (2)	5.43105, 5.43105, 5.43105
α, β, γ (°)	90, 90, 90	90, 90, 90
V (Å^3)	1001.66 (3)	160.20
Z	4	8
Radiation type	$K\alpha_1, K\alpha_2, \lambda = 1.540629, 1.544451$ Å	$K\alpha_1, K\alpha_2, \lambda = 1.540629, 1.544451$ Å
Specimen shape, size (mm)	Flat sheet, 24 × 24	Flat sheet, 24 × 24
Data collection		
Diffractometer	Bruker D2 Phaser	Bruker D2 Phaser
Specimen mounting	Normal sample holder with Kapton film	Normal sample holder with Kapton film
Data collection mode	Reflection	Reflection
Scan method	Step	Step
2θ values (°)	$2\theta_{min} = 4.908, 2\theta_{max} = 69.916, 2\theta_{step} = 0.020$	$2\theta_{min} = 4.908, 2\theta_{max} = 69.916, 2\theta_{step} = 0.020$
Refinement		
R factors and goodness of fit	$R_p = 0.038, R_{wp} = 0.049, R_{exp} = 0.034, R(F^2) = 0.059, \chi^2 = 2.103$	$R_p = 0.038, R_{wp} = 0.049, R_{exp} = 0.034, R(F^2) = 0.059, \chi^2 = 2.103$
No. of parameters	73	73
No. of restraints	29	29

The same symmetry and lattice parameters were used for the DFT calculation. Computer programs: *DIFFRAC.Measurement* (Bruker, 2009), *FOX* (Favre-Nicolin & Černý, 2002), *GSAS* (Larson & Von Dreele, 2004), *EXPGUI* (Toby, 2001), *DIAMOND* (Crystal Impact, 2015) and *publCIF* (Westrip, 2010).

3. Supramolecular features

The $[KO_n]$ coordination polyhedra share edges and corners to form a three-dimensional framework (Fig. 3), with channels running down the c axis. The only hydrogen bond is an intramolecular one (Table 1) involving the hydroxy group and the central carboxylate group, with graph-set motif $S(5)$. The Mulliken overlap population in the hydrogen-acceptor bond is 0.076 e. By the correlation in Rammohan & Kaduk (2016a), this hydrogen bond accounts for 15.1 kcal per mole of crystal energy.

4. Database survey

Details of the comprehensive literature search for citrate structures are presented in Rammohan & Kaduk (2016a). A reduced-cell search of the cell of anhydrous tripotassium citrate in the Cambridge Structural Database (Groom *et al.*, 2016) (increasing the default tolerance from 1.5 to 2.0%) yielded 208 hits, but limiting the chemistry to C, H, K, and O only resulted in no hits. The powder pattern is now contained in the the Powder Diffraction File (ICDD, 2015) as entry 00-064-1370.

5. Synthesis and crystallization

Potassium citrate monohydrate was synthesized by dissolving 2.0796 g (10.0 mmole) $H_3C_6H_5O_7(H_2O)$ in 20 ml deionized water. 2.0731g K_2CO_3 (15.0 mmole, Sigma-Aldrich) was added to the citric acid solution slowly with stirring. The resulting clear colourless solution was evaporated to dryness in a 333 K oven. The powder pattern matched PDF entry 02-064-1651, confirming the structure as potassium citrate monohydrate (Carrell *et al.*, 1987). The monohydrate was heated at 15 K min^{-1} to 498 K, and held there for two minutes (the white solid started to discolour). The white solid was removed from the oven, and immediately placed in a sealed glass jar to cool.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. The white solid was ground and blended with NIST SRM 640b Si internal standard in a mortar and pestle. The specimen was protected from the atmosphere by an 8 micron Kapton film attached to the sample holder with petroleum jelly. (The sample hydrates slowly on contact with ambient atmosphere.)

The pattern (Fig. 4) was indexed on a primitive ortho-rhombic unit cell using ITO (Visser, 1969). Manual examination of the systematic absences suggested space group $Pna2_1$. 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 solved with *FOX* (Favre-Nicolin &
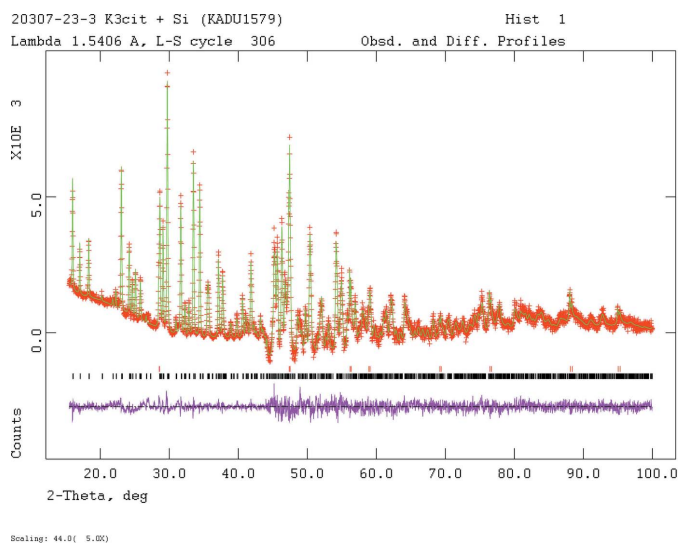
Figure 4
Rietveld plot for the refinement of $K_3C_6H_5O_7$. The red crosses represent the observed data points, and the green line is the calculated pattern. The magenta curve is the difference pattern, plotted at the same scale as the other patterns. The vertical scale for angles > 44.0° has been multiplied by a factor of 5. The lower row of black tick marks indicates the reflection positions for the major phase and the upper row of red tick marks is for the silicon internal standard.

Černý, 2002) using a citrate moiety and three potassium atoms as fragments. The structure was refined by the Rietveld method using *GSAS/EXPGUI* (Larson & Von Dreele, 2004; Toby, 2001). All C—C and C—O bond lengths were restrained, as were all bond angles. The hydrogen atoms were included at fixed positions, which were recalculated during the course of the refinement using *Materials Studio* (Dassault Systemes, 2014). The U_{iso} values of the atoms in the central and outer portions of the citrate anion were constrained to be equal, and the U_{iso} values of the hydrogen atoms were constrained to be 1.3× those of the atoms to which they are attached.

The ADDSYM module of *PLATON* (Spek, 2009) suggested the presence of an additional centre of symmetry, and that the space group was *Pnam*. Refinement in this space group yielded poorer residuals, so we believe that *Pna2$_1$* is the correct space group.

7. DFT calculations

After the Rietveld refinement, a density functional geometry optimization (fixed experimental unit cell) was carried out using *CRYSTAL09* (Dovesi *et al.*, 2005). The basis sets for the C, H, and O atoms were those of Gatti *et al.* (1994), and the basis set for K was that of Dovesi *et al.* (1991). The calculation used 8 *k*-points and the B3LYP functional, and took about 66 h on a 2.4 GHz PC. The U_{iso} values from the Rietveld refinement were assigned to the optimized fractional coordinates.

References

Bravais, A. (1866). In *Etudes Cristallographiques.* Paris: Gauthier Villars.
Bruker (2009). *DIFFRAC.Measurement*. Bruker AXS Inc., Madison, Wisconsin, USA.
Carrell, H. L., Glusker, J. P., Piercy, E. A., Stallings, W. C., Zacharias, D. E., Davis, R. L., Astbury, C. & Kennard, C. H. L. (1987). *J. Am. Chem. Soc.* **109**, 8067–8071.
Crystal Impact (2015). *DIAMOND*. Crystal Impact GbR, Bonn, Germany. http://www.crystalimpact.com/diamond.
Dassault Systemes (2014). *Materials Studio*. BIOVIA, San Diego, California, USA.
Donnay, J. D. H. & Harker, D. (1937). *Am. Mineral.* **22**, 446–467.
Dovesi, R., Orlando, R., Civalleri, B., Roetti, C., Saunders, V. R. & Zicovich-Wilson, C. M. (2005). *Z. Kristallogr.* **220**, 571–573.
Dovesi, R., Roetti, C., Freyria-Fava, C., Prencipe, M. & Saunders, V. R. (1991). *Chem. Phys.* **156**, 11–19.
Favre-Nicolin, V. & Černý, R. (2002). *J. Appl. Cryst.* **35**, 734–743.
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.
Gatti, C., Saunders, V. R. & Roetti, C. (1994). *J. Chem. Phys.* **101**, 10686–10696.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Howard, C. J. (1982). *J. Appl. Cryst.* **15**, 615–620.
ICDD (2015). *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). Private communication (No. 1446457–1446458). CCDC, Cambridge, England.
Kaduk, J. A. & Stern, C. (2016b). Private communication (No. 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.
Rammohan, A. & Kaduk, J. A. (2016a). *Acta Cryst.* B**72**. Submitted.
Rammohan, A. & Kaduk, J. A. (2016b). *Acta Cryst.* E**72**, 170–173.
Rammohan, A. & Kaduk, J. A. (2016c). *Acta Cryst.* E**72**, 403–406.
Rammohan, A. & Kaduk, J. A. (2016d). *Acta Cryst.* E**72**, 793–796.
Rammohan, A. & Kaduk, J. A. (2016e). *Acta Cryst.* E**72**, 854–857.
Rammohan, A. Sarjeant, A. A. & Kaduk, J. A. (2016). *Acta Cryst.* E**72**, 943–946.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Stephens, P. W. (1999). *J. Appl. Cryst.* **32**, 281–289.
Streek, J. van de & Neumann, M. A. (2014). *Acta Cryst.* B**70**, 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.

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Computing details

Data collection: *DIFFRAC.Measurement* (Bruker, 2009) for KADU1578_phase_1. Program(s) used to solve structure: *FOX* (Favre-Nicolin & Černý, 2002) for KADU1578_phase_1. Program(s) used to refine structure: *GSAS* (Larson & Von Dreele, 2004) and *EXPGUI* (Toby, 2001) for KADU1578_phase_1. Molecular graphics: *DIAMOND* (Crystal Impact, 2015) for KADU1578_phase_1. Software used to prepare material for publication: *publCIF* (Westrip, 2010) for KADU1578_phase_1.

(KADU1578_phase_1) Poly[(μ-2-hydroxypropane-1,2,3-tricarboxylato0tripotassium]

Crystal data

[$K_3(C_6H_5O_7)$]	$c = 10.4241$ (2) Å
$M_r = 306.39$	$V = 1001.66$ (3) Å^3
Orthorhombic, $Pna2_1$	$Z = 4$
$a = 7.7062$ (2) Å	$T = 300$ K
$b = 12.4693$ (3) Å	

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.841 (4)	0.883 (3)	0.28611	0.023 (3)*
C2	0.926 (4)	0.915 (3)	0.411 (2)	0.021 (5)*
C3	0.814 (2)	0.8966 (12)	0.532 (3)	0.021 (5)*
C4	0.937 (4)	0.910 (4)	0.647 (3)	0.021 (5)*
C5	0.857 (4)	0.872 (3)	0.771 (3)	0.023 (3)*
C6	0.6539 (18)	0.9724 (11)	0.541 (5)	0.023 (3)*
H7	1.04032	0.90124	0.41652	0.027 (7)*
H8	0.9082	0.99396	0.3928	0.027 (7)*
H9	0.97168	0.98330	0.645913	0.027 (7)*
H10	1.01382	0.84403	0.63413	0.027 (7)*
O11	0.695 (4)	0.9217 (19)	0.252 (4)	0.023 (3)*
O12	0.936 (4)	0.816 (2)	0.226 (4)	0.023 (3)*
O13	0.935 (4)	0.821 (2)	0.859 (4)	0.023 (3)*
O14	0.697 (4)	0.894 (2)	0.787 (4)	0.023 (3)*
O15	0.6828 (17)	1.0726 (11)	0.529 (6)	0.023 (3)*
O16	0.5076 (16)	0.9267 (10)	0.533 (5)	0.023 (3)*
O17	0.7473 (14)	0.7877 (11)	0.525 (5)	0.023 (3)*

H18	0.6543	0.8072	0.525	0.030 (4)*
K19	0.355 (3)	0.8827 (13)	0.743 (3)	0.0442 (17)*
K20	0.1336 (10)	0.7108 (4)	0.021 (3)	0.0442 (17)*
K21	0.148 (3)	0.3753 (14)	0.797 (3)	0.0442 (17)*

Geometric parameters (Å, º)

C1—C2	1.510 (5)	O13—K20[viii]	2.90 (3)
C1—O11	1.278 (9)	O14—C5	1.270 (9)
C1—O12	1.271 (9)	O14—O13	2.18 (3)
C2—C1	1.510 (5)	O14—K19	2.68 (4)
C2—C3	1.544 (4)	O14—K20[viii]	2.81 (3)
C2—H7	0.90 (3)	O14—K21[ii]	2.90 (3)
C2—H8	1.01 (4)	O15—C6	1.275 (8)
C3—C2	1.544 (4)	O15—K19[i]	3.05 (6)
C3—C4	1.544 (5)	O15—K20[ix]	2.987 (15)
C3—C6	1.553 (4)	O15—K20[x]	3.051 (15)
C3—O17	1.452 (8)	O15—K21[ii]	2.88 (5)
C4—C3	1.544 (5)	O16—C6	1.266 (8)
C4—C5	1.511 (5)	O16—K19	2.55 (4)
C4—H9	0.95 (4)	O16—K21[iii]	2.81 (4)
C4—H10	1.03 (5)	O17—C3	1.452 (8)
C5—C4	1.511 (5)	O17—H18	0.757 (11)
C5—O13	1.268 (9)	O17—K19[ii]	3.22 (4)
C5—O14	1.270 (9)	O17—K21[vi]	3.23 (4)
C6—C3	1.553 (4)	H18—O17	0.757 (11)
C6—O15	1.275 (8)	K19—O11[x]	2.47 (2)
C6—O16	1.266 (8)	K19—O13[xi]	2.88 (3)
H7—C2	0.90 (3)	K19—O14	2.68 (4)
H8—C2	1.01 (4)	K19—O15[x]	3.05 (6)
H9—C4	0.82 (4)	K19—O16	2.55 (4)
H10—C4	1.03 (5)	K19—O17[xi]	3.22 (4)
O11—C1	1.278 (9)	K20—O11[xi]	2.96 (3)
O11—K19[i]	2.47 (2)	K20—O12[xii]	2.93 (3)
O11—K20[ii]	2.96 (3)	K20—O12[xi]	3.18 (3)
O11—K21[iii]	2.75 (4)	K20—O13[xiii]	2.66 (3)
O12—C1	1.271 (9)	K20—O13[xiv]	2.90 (3)
O12—K20[iv]	2.93 (3)	K20—O14[xiv]	2.81 (3)
O12—K20[ii]	3.18 (3)	K20—O15[xv]	2.987 (15)
O12—K21[v]	3.37 (4)	K20—O15[i]	3.051 (15)
O12—K21[vi]	2.58 (3)	K21—O11[xvi]	2.75 (4)
O13—C5	1.268 (9)	K21—O12[xvii]	2.58 (3)
O13—O14	2.18 (3)	K21—O14[xi]	2.90 (3)
O13—K19[iv]	3.54 (4)	K21—O15[xi]	2.88 (5)
O13—K19[ii]	2.88 (3)	K21—O16[xvi]	2.81 (4)
O13—K20[vii]	2.66 (3)	K21—O17[xvii]	3.23 (4)
C2—C1—O11	121 (2)	O11[x]—K19—O13[xi]	152.6 (13)

C2—C1—O12	111 (2)	O11[x]—K19—O14	95.5 (9)
O11—C1—O12	128 (2)	O11[x]—K19—O15[x]	76.6 (11)
C1—C2—C3	115 (2)	O11[x]—K19—O16	83.8 (8)
C2—C3—C4	106.0 (13)	O11[x]—K19—O17[xi]	129.6 (11)
C2—C3—C6	114 (3)	O13[xi]—K19—O14	76.3 (11)
C2—C3—O17	107 (2)	O13[xi]—K19—O15[x]	76.7 (7)
C4—C3—C6	112 (2)	O13[xi]—K19—O16	117.0 (10)
C4—C3—O17	111 (2)	O13[xi]—K19—O17[xi]	76.7 (9)
C6—C3—O17	107.1 (12)	O14—K19—O15[x]	85.2 (9)
C3—C4—C5	112 (2)	O14—K19—O16	71.5 (11)
C4—C5—O13	126 (2)	O14—K19—O17[xi]	114.2 (10)
C4—C5—O14	116 (2)	O15[x]—K19—O16	147.9 (10)
O13—C5—O14	118 (2)	O15[x]—K19—O17[xi]	141.9 (9)
C3—C6—O15	116.9 (13)	O16—K19—O17[xi]	69.7 (7)
C3—C6—O16	115.3 (13)	O11[xi]—K20—O12[xii]	74.9 (9)
O15—C6—O16	126.2 (16)	O11[xi]—K20—O12[xi]	43.7 (5)
C1—O11—K19[i]	122 (3)	O11[xi]—K20—O13[xiii]	153.7 (8)
C1—O11—K20[ii]	98.9 (17)	O11[xi]—K20—O13[xiv]	105.6 (8)
C1—O11—K21[iii]	136 (3)	O11[xi]—K20—O14[xiv]	114.7 (5)
K19[i]—O11—K20[ii]	119.7 (12)	O11[xi]—K20—O15[xv]	77.6 (12)
K19[i]—O11—K21[iii]	93.6 (11)	O11[xi]—K20—O15[i]	113.4 (13)
K20[ii]—O11—K21[iii]	82.3 (8)	O12[xii]—K20—O12[xi]	86.3 (8)
C1—O12—K20[iv]	162 (3)	O12[xii]—K20—O13[xiii]	86.2 (5)
C1—O12—K20[ii]	88.7 (17)	O12[xii]—K20—O13[xiv]	154.3 (8)
C1—O12—K21[vi]	109 (3)	O12[xii]—K20—O14[xiv]	158.6 (7)
K20[iv]—O12—K20[ii]	81.0 (7)	O12[xii]—K20—O15[xv]	79.2 (12)
K20[iv]—O12—K21[vi]	85.8 (9)	O12[xii]—K20—O15[i]	80.0 (10)
K20[ii]—O12—K21[vi]	84.9 (10)	O12[xi]—K20—O13[xiii]	154.4 (9)
C5—O13—K19[ii]	92 (2)	O12[xi]—K20—O13[xiv]	77.9 (4)
C5—O13—K20[vii]	172.2 (18)	O12[xi]—K20—O14[xiv]	114.1 (8)
C5—O13—K20[viii]	96.3 (17)	O12[xi]—K20—O15[xv]	121.3 (12)
K19[ii]—O13—K20[vii]	86.2 (9)	O12[xi]—K20—O15[i]	74.6 (10)
K19[ii]—O13—K20[viii]	87.4 (10)	O13[xiii]—K20—O13[xiv]	99.4 (7)
K20[vii]—O13—K20[viii]	91.3 (7)	O13[xiii]—K20—O14[xiv]	77.9 (8)
C5—O14—K19	157 (3)	O13[xiii]—K20—O15[xv]	81.1 (10)
C5—O14—K20[viii]	100.5 (18)	O13[xiii]—K20—O15[i]	80.1 (10)
C5—O14—K21[ii]	110 (3)	O13[xiv]—K20—O14[xiv]	44.9 (6)
K19—O14—K20[viii]	87.4 (8)	O13[xiv]—K20—O15[xv]	126.3 (12)
K19—O14—K21[ii]	86.0 (11)	O13[xiv]—K20—O15[i]	76.4 (9)
K20[viii]—O14—K21[ii]	114.1 (12)	O14[xiv]—K20—O15[xv]	84.2 (14)
C6—O15—K19[i]	105 (4)	O14[xiv]—K20—O15[i]	110.8 (11)
C6—O15—K20[ix]	115.2 (11)	O15[xv]—K20—O15[i]	152.7 (6)
C6—O15—K20[x]	162.1 (14)	O11[xvi]—K21—O12[xvii]	84.5 (12)
C6—O15—K21[ii]	96 (4)	O11[xvi]—K21—O14[xi]	84.9 (8)
K19[i]—O15—K20[ix]	77.8 (11)	O11[xvi]—K21—O15[xi]	82.9 (9)
K19[i]—O15—K20[x]	81.7 (12)	O11[xvi]—K21—O16[xvi]	71.9 (10)
K19[i]—O15—K21[ii]	154.0 (5)	O11[xvi]—K21—O17[xvii]	119.9 (10)
K20[ix]—O15—K20[x]	82.4 (3)	O12[xvii]—K21—O14[xi]	160.0 (12)

K20ⁱˣ—O15—K21ⁱⁱ	79.7 (11)	O12ˣᵛⁱⁱ—K21—O15ˣⁱ	87.5 (7)
K20ˣ—O15—K21ⁱⁱ	82.5 (12)	O12ˣᵛⁱⁱ—K21—O16ˣᵛⁱ	110.7 (10)
C6—O16—K19	117 (3)	O12ˣᵛⁱⁱ—K21—O17ˣᵛⁱⁱ	72.0 (9)
C6—O16—K21ⁱⁱⁱ	123 (3)	O14ˣⁱ—K21—O15ˣⁱ	74.4 (10)
K19—O16—K21ⁱⁱⁱ	120.5 (5)	O14ˣⁱ—K21—O16ˣᵛⁱ	81.9 (8)
C3—O17—H18	91.9 (14)	O14ˣⁱ—K21—O17ˣᵛⁱⁱ	128.0 (11)
C3—O17—K19ⁱⁱ	119 (2)	O15ˣⁱ—K21—O16ˣᵛⁱ	146.8 (10)
C3—O17—K21ᵛⁱ	123 (2)	O15ˣⁱ—K21—O17ˣᵛⁱⁱ	146.4 (9)
K19ⁱⁱ—O17—K21ᵛⁱ	92.3 (3)	O16ˣᵛⁱ—K21—O17ˣᵛⁱⁱ	66.8 (7)

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

(KADU1578_phase_2) Poly[(µ₁₄-2-hydroxypropane-1,2,3-tricarboxylato0tripotassium]

Crystal data

Si	$V = 160.20$ Å³
$M_r = 28.09$	$Z = 8$
Cubic, $Fd\bar{3}m$	$T = 300$ K
$a = 5.43105$ Å	

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

	x	y	z	U_{iso}*/U_{eq}
Si1	0.125	0.125	0.125	0.01*

Geometric parameters (Å, °)

Si1—Si1ⁱ	2.3517	Si1—Si1ⁱⁱⁱ	2.3517
Si1—Si1ⁱⁱ	2.3517	Si1—Si1ⁱᵛ	2.3517
Si1ⁱ—Si1—Si1ⁱⁱ	109.4712	Si1ⁱⁱ—Si1—Si1ⁱⁱⁱ	109.4712
Si1ⁱ—Si1—Si1ⁱⁱⁱ	109.4712	Si1ⁱⁱ—Si1—Si1ⁱᵛ	109.4712
Si1ⁱ—Si1—Si1ⁱᵛ	109.4712	Si1ⁱⁱⁱ—Si1—Si1ⁱᵛ	109.4712

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

(kadu1578_DFT)

Crystal data

[K₃(C₆H₅O₇)]	$c = 10.4222$ (3) Å
$M_r = 306.37$	$V = 1001.66$ Å³
Orthorhombic, $PNA2_1$	$Z = 4$
$a = 7.7074$ (2) Å	Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
$b = 12.4676$ (3) Å	$T = 300$ K

Data collection

Density functional calculation	$k = \rightarrow$
$h = \rightarrow$	$l = \rightarrow$

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.83999	0.87608	0.28090	0.03110*
C2	0.91941	0.91369	0.40840	0.04090*
C3	0.80507	0.89495	0.52759	0.04090*
C4	0.91865	0.91285	0.64734	0.04090*
C5	0.83786	0.87569	0.77456	0.03110*
C6	0.64533	0.97237	0.52736	0.03110*
H7	1.04170	0.87092	0.42230	0.05310*
H8	0.94795	0.99967	0.40314	0.05310*
H9	0.94862	0.99862	0.65281	0.05310*
H10	1.04015	0.86912	0.63386	0.05310*
O11	0.69006	0.90966	0.25219	0.03110*
O12	0.93097	0.81435	0.21142	0.03110*
O13	0.92734	0.81335	0.84433	0.03110*
O14	0.68837	0.91019	0.80291	0.03110*
O15	0.67248	1.07226	0.52772	0.03110*
O16	0.49925	0.92599	0.52665	0.03110*
O17	0.74146	0.78645	0.52713	0.03110*
H18	0.61565	0.79949	0.52593	0.04040*
K19	0.35417	0.87929	0.75053	0.04550*
K20	0.13670	0.70910	0.02740	0.04550*
K21	0.14595	0.37878	0.80253	0.04550*

Bond lengths (Å)

C1—C2	1.536	C4—C5	1.536
C1—O11	1.265	C4—H9	1.096
C1—O12	1.268	C4—H10	1.093
C2—C3	1.541	C5—O13	1.268
C2—H7	1.093	C5—O14	1.265
C2—H8	1.096	C6—O15	1.263
C3—C4	1.541	C6—O16	1.266
C3—C6	1.564	O17—H18	0.983
C3—O17	1.439		

Hydrogen-bond geometry (Å, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O17—H18⋯O16	0.983	1.814	2.552	129.1