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Sodium potassium hydrogen citrate, NaKHC₆H₅O₇

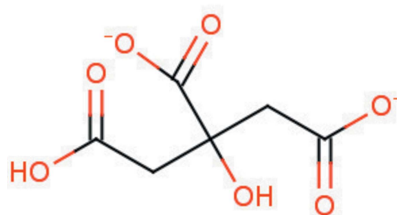
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The crystal structure of sodium potassium hydrogen citrate has been solved and refined using laboratory X-ray powder diffraction data, and optimized using density functional theory techniques. The Na⁺ cation is six-coordinate, with a bond-valence sum of 1.17. The K⁺ cation is also six-coordinate, with a bond-valence sum of 1.08. The distorted [NaO₆] octahedra share edges, forming chains along the *a* axis. The likewise distorted [KO₆] octahedra share edges with the [NaO₆] octahedra on either side of the chain, and share corners with other [KO₆] octahedra, resulting in triple chains along the *a* axis. The most prominent feature of the structure is the chain along [111] of very short, very strong hydrogen bonds; the O···O distances are 2.414 and 2.400 Å. The Mulliken overlap populations in these hydrogen bonds are 0.138 and 0.142 e, which correspond to hydrogen-bond energies of 20.3 and 20.6 kcal mol⁻¹.

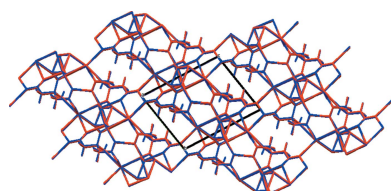
1. Chemical context

We have carried out 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. Most of the new structures were solved using powder diffraction data (laboratory and/or synchrotron), but single crystals were used where available. The general trends and conclusions about the 16 new compounds and 12 previously characterized structures are being reported separately (Rammohan & Kaduk, 2015). The initial study considered salts containing one type of Group 1 cation. The title compound (Fig. 1) represents the beginning of an extension of the study to salts containing more than one alkali metal cation.



2. Structural commentary

The root-mean-square deviation of the non-hydrogen atoms in the refined and optimized structures is only 0.088 Å. A comparison of the refined and optimized structures is given in Fig. 2. The excellent agreement between the structures is strong evidence that the experimental structure is correct (van de Streek & Neumann, 2014). This discussion uses the DFT-



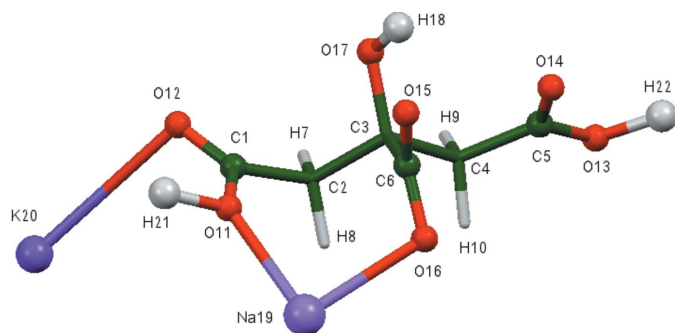


Figure 1
The asymmetric unit, with the atom numbering and 50% probability spheroids.

optimized structure. Most of the bond lengths, and all of the bond angles and torsion angles fall within the normal ranges indicated by a *Mercury* Mogul Geometry Check (Macrae *et al.*, 2008). Only the C6–O15 [observed = 1.281 (4), optimized = 1.268, normal = 1.20 (2) Å, Z-score = 2.7] and C1–O11 [observed = 1.260 (4), optimized = 1.318, normal = 1.330 (3) Å, Z-score = 3.9] bonds are flagged as unusual. 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. The central carboxylate group and the hydroxyl group occur in the normal planar arrangement. The citrate chelates to Na19 through the terminal carboxylate oxygen O11 and the central carboxylate oxygen O16. The Na⁺ cation is six-coordinate, with a bond-valence sum of 1.17. The K⁺ cation is also six-coordinate, with a bond-valence sum of 1.08. Both cations are thus slightly crowded. The metal–oxygen bonding is ionic, based on the Mulliken overlap populations.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) morphology suggests that we might expect platy morphology for sodium potassium hydrogen citrate, with {001} as the principal faces. A 4th-order spherical harmonic preferred orientation model was included in the refinement; the texture index was only 1.013, indicating that preferred orientation was not significant in this rotated flat-plate specimen. The powder pattern is included in the Powder Diffraction File as entry 00-065-1255.

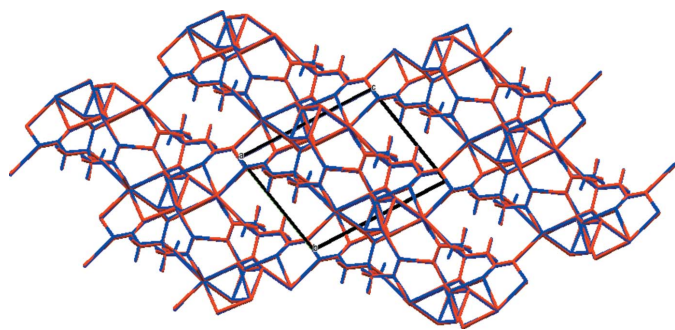


Figure 2
Comparison of the refined and optimized structures of sodium potassium hydrogen citrate. The refined structure is in red, and the DFT-optimized structure is in blue.

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

<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>	Overlap
O11–H21···O11 ⁱ	1.207	1.207	2.414	180.0	0.138
O13–H22···O13 ⁱⁱ	1.200	1.200	2.400	180.0	0.142
O17–H18···O15	0.971	2.179	2.676	110.3	0.033
O17–H18···O11 ⁱⁱⁱ	0.971	2.227	3.060	143.1	0.028

Symmetry codes: (i) 1 – *x*, 1 – *y*, 1 – *z*; (ii) 2 – *x*, 2 – *y*, 2 – *z*; (iii) 1 + *x*, *y*, *z*.

3. Supramolecular features

In the crystal structure (Fig. 3), distorted [NaO₆] octahedra share edges to form chains along the *a* axis. The likewise distorted [KO₆] octahedra share edges with the [NaO₆] octahedra on either side of the chain, and share corners with other [KO₆] octahedra, resulting in triple chains along the *a* axis. The most prominent feature of the structure is the chain along [111] of very short, very strong O–H···O hydrogen bonds (Table 1); the refined O···O distances are 2.385 (15) and 2.346 (14) Å, and the optimized O···O distances are 2.414 and 2.400 Å. The Mulliken overlap populations in these hydrogen bonds are 0.138 and 0.142 e, which correspond to hydrogen bond energies of 20.3 and 20.6 kcal mol^{–1}. The distances indicate that these are among the shortest O–H···O hydrogen bonds ever reported. H18 forms bifurcated hydrogen bonds; one is intramolecular to O15, and the other intermolecular to O11.

4. Database survey

Details of the comprehensive literature search for citrate structures are presented in Rammohan & Kaduk (2015). A reduced cell search in the Cambridge Structural Database (Groom & Allen, 2014) (increasing the default tolerance from 1.5 to 2.0%, to account for the differences between ambient and low-temperature lattice parameters) yielded 35 hits, but limiting the chemistry to C, H, O, Na, and K only resulted in no hits. The powder pattern matched no entry in the Powder Diffraction File (ICDD, 2015).

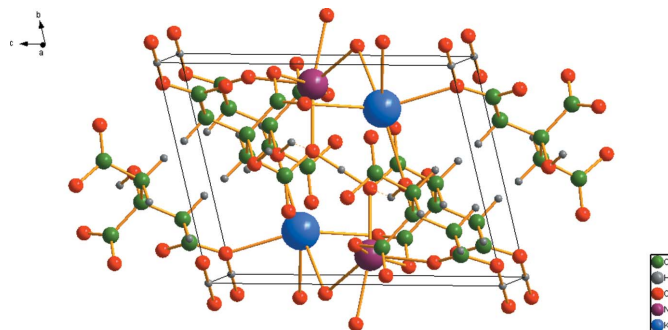


Figure 3
Crystal structure of NaKHC₆H₅O₇, viewed approximately down the *a* axis.

5. Synthesis and crystallization

2.0832 g (10.0 mmol) $\text{H}_3\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})$ was dissolved in 10 mL deionized water. 0.5282 g Na_2CO_3 (10.0 mmol Na, Sigma–Aldrich) and 0.6913 g K_2CO_3 (10.0 mmol, Sigma–Aldrich) were added to the citric acid solution slowly with stirring. The resulting clear colourless solution was evaporated to dryness in a 393 K oven.

6. Refinement details

The powder pattern (Fig. 4) was indexed using *Jade 9.5* (MDI, 2012). Pseudovoigt profile coefficients were as parameterized in Thompson *et al.* (1987) and the asymmetry correction of Finger *et al.* (1994) was applied and microstrain broadening by Stephens (1999). The structure was solved with *FOX* (Favre-Nicolin & Černý, 2002) using a citrate, Na, and K as fragments. Two of the 10 solutions yielded much lower cost functions than the others. Centrosymmetric pairs of close O···O contacts made it clear that H21 and H22 were located on centers of symmetry between these oxygen atoms, forming very strong hydrogen bonds. The hydrogen atoms were included at fixed positions, which were re-calculated during the course of the refinement. Crystal data, data collection and structure refinement details are summarized in Table 2. The U_{iso} of C2, C3, and C4 were constrained to be equal, and those of H7, H8, H9, and H10 were constrained to be $1.3\times$ that of these carbon atoms. The U_{iso} of C1, C5, C6, and the oxygen atoms were constrained to be equal, and that of H18 was constrained to be $1.3\times$ this value. The U_{iso} of H21 and H22 were fixed.

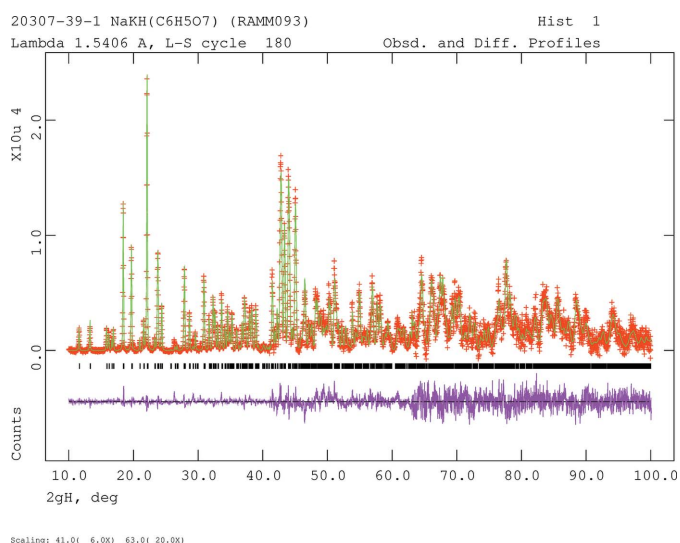


Figure 4

Rietveld plot for the refinement of $\text{NaKH}(\text{C}_6\text{H}_5\text{O}_7)$. 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 has been multiplied by a factor of 6 for $2\theta > 41.0^\circ$, and by a factor of 20 for $2\theta > 63.0^\circ$. The row of black tick marks indicates the reflection positions for the phase.

Table 2

Experimental details.

Powder data	
Crystal data	
Chemical formula	$\text{NaK}(\text{C}_6\text{H}_5\text{O}_7)$
M_r	252.19
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	300
a, b, c (Å)	5.99933 (18), 8.2277 (2), 10.1419 (3)
α, β, γ (°)	74.8964 (19), 76.019 (2), 71.4496 (14)
V (Å ³)	451.27 (3)
Z	2
Radiation type	$K\alpha_1, K\alpha_2, \lambda = 1.540629, 1.544451$ Å
Specimen shape, size (mm)	Flat sheet, 24×24
Data collection	
Diffractometer	Bruker D2 Phaser
Specimen mounting	Standard holder
Data collection mode	Reflection
Data collection method	Step
θ values (°)	$2\theta_{\text{min}} = 4.908$ $2\theta_{\text{max}} = 99.914$ $2\theta_{\text{step}} = 0.020$
Refinement	
R factors and goodness of fit	$R_p = 0.034, R_{\text{wp}} = 0.046,$ $R_{\text{exp}} = 0.024, R(F^2) = 0.08172,$ $\chi^2 = 4.040$
No. of data points	4452
No. of parameters	98
No. of restraints	29
H-atom treatment	Only H-atom displacement parameters refined

The same symmetry and lattice parameters were used for the DFT calculation. Computer programs: *DIFFRAC.Measurement* (Bruker, 2009), *PowDLL* (Kourkoumelis, 2013), *FOX* (Favre-Nicolin & Černý, 2002), *GSAS* (Larson & Von Dreele, 2004), *EXPGUI* (Toby, 2001), *DIAMOND* (Putz & Brandenburg, 2015), *publCIF* (Westrip, 2010).

6.1. Density functional geometry optimization

A density functional geometry optimization (fixed experimental unit cell) was carried out using *CRYSTAL09* (Dovesi *et al.*, 2005). The basis sets for the H, C, and O atoms were those of Gatti *et al.* (1994), the basis sets for Na and K were those of Dovesi *et al.* (1991). The calculation used 8 k-points and the B3LYP functional, and took about 42 h on a 2.8 GHz PC. The observed U_{iso} were assigned to the refined values.

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supporting information

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Sodium potassium hydrogen citrate, NaKHC₆H₅O₇

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Computing details

Data collection: *DIFFRAC.Measurement* (Bruker, 2009) for RAMM093_publ. Data reduction: *PowDLL* (Kourkoumelis, 2013) for RAMM093_publ. Program(s) used to solve structure: *FOX* (Favre-Nicolin & Černý, 2002) for RAMM093_publ. Program(s) used to refine structure: *GSAS* (Larson & Von Dreele, 2004), *EXPGUI* (Toby, 2001) for RAMM093_publ. Molecular graphics: *DIAMOND* (Putz & Brandenburg, 2015) for RAMM093_publ. Software used to prepare material for publication: *publCIF* (Westrip, 2010) for RAMM093_publ.

(RAMM093_publ) sodium potassium hydrogen citrate

Crystal data

NaK(C₆H₅O₇)

$M_r = 252.19$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.99933$ (18) Å

$b = 8.2277$ (2) Å

$c = 10.1419$ (3) Å

$\alpha = 74.8964$ (19)°

$\beta = 76.019$ (2)°

$\gamma = 71.4496$ (14)°

$V = 451.27$ (3) Å³

$Z = 2$

$D_x = 1.864$ Mg m⁻³

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

$T = 300$ K

white

flat sheet, 24 × 24 mm

Specimen preparation: Prepared at 393 K and 101 kPa

Data collection

Bruker D2 Phaser
diffractometer

Radiation source: sealed X-ray tube

Ni filter monochromator

Specimen mounting: standard holder

Data collection mode: reflection

Scan method: step

$2\theta_{\min} = 4.908^\circ$, $2\theta_{\max} = 99.914^\circ$, $2\theta_{\text{step}} = 0.020^\circ$

Refinement

Least-squares matrix: full

$R_p = 0.034$

$R_{\text{wp}} = 0.046$

$R_{\text{exp}} = 0.024$

$R(F^2) = 0.08172$

$\chi^2 = 4.040$

Profile function: CW Profile function number 4 with 27 terms Pseudovoigt profile coefficients as parameterized in Thompson *et al.* (1987). Asymmetry correction of Finger *et al.* (1994). Microstrain broadening by Stephens (1999). #1(GU) = 2.580 #2(GV) = 0.000 #3(GW) = 1.999 #4(GP) = 0.000 #5(LX) = 4.774 #6(ptec) = 0.64 #7(trns) = 4.34 #8(shft) = 4.0539 #9(sfec) = 0.00 #10(S/L) = 0.0168 #11(H/L) = 0.0200 #12(eta) = 0.0000 Peak tails are ignored where the intensity is below 0.0050 times the peak Aniso. broadening axis 0.0 0.0 1.0

98 parameters

29 restraints

2 constraints

Only H-atom displacement parameters refined
Weighting scheme based on measured s.u.'s
(Δ/σ)_{max} = 0.04

Background function: GSAS Background
function number 1 with 10 terms. Shifted
Chebyshev function of 1st kind 1: 1400.27 2:
-1034.48 3: 405.201 4: -101.434 5: 48.9076 6:
-20.5280 7: -17.7840 8: 47.9002 9: -30.1492 10:
17.3246

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	0.6011 (16)	0.4794 (8)	0.6851 (9)	0.0215 (12)*
C2	0.5815 (15)	0.5669 (11)	0.8023 (8)	0.003 (3)*
C3	0.7804 (11)	0.6586 (7)	0.7744 (6)	0.003 (3)*
C4	0.7535 (15)	0.7282 (10)	0.9061 (7)	0.003 (3)*
C5	0.9060 (17)	0.8477 (14)	0.8955 (7)	0.0215 (12)*
C6	0.7448 (13)	0.8129 (8)	0.6491 (6)	0.0215 (12)*
H7	0.59718	0.46465	0.90250	0.003 (4)*
H8	0.40047	0.66779	0.81496	0.003 (4)*
H9	0.80365	0.61352	0.99492	0.003 (4)*
H10	0.55837	0.80168	0.93544	0.003 (4)*
O11	0.5025 (16)	0.5759 (9)	0.5848 (8)	0.0215 (12)*
O12	0.6312 (14)	0.3154 (9)	0.7090 (7)	0.0215 (12)*
O13	0.9018 (14)	0.8943 (10)	1.0079 (7)	0.0215 (12)*
O14	1.0287 (14)	0.9043 (10)	0.7803 (7)	0.0215 (12)*
O15	0.9008 (15)	0.8051 (10)	0.5379 (7)	0.0215 (12)*
O16	0.5507 (13)	0.9325 (9)	0.6540 (6)	0.0215 (12)*
O17	1.0087 (13)	0.5402 (9)	0.7419 (7)	0.0215 (12)*
H18	1.10980	0.60623	0.68105	0.0279 (16)*
Na19	0.2588 (11)	0.8708 (7)	0.5423 (6)	0.051 (3)*
K20	0.1831 (8)	0.1991 (5)	0.7186 (3)	0.0406 (16)*
H21	0.5	0.5	0.5	0.03*
H22	1.0	1.0	1.0	0.03*

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

C1—C2	1.507 (2)	O14—Na19 ⁱⁱ	2.510 (9)
C1—O11	1.260 (4)	O14—K20 ^{iv}	2.737 (8)
C1—O12	1.268 (4)	O15—C6	1.281 (4)
C2—C1	1.507 (2)	O15—Na19 ⁱⁱ	2.388 (9)
C2—C3	1.540 (2)	O15—Na19 ^v	2.512 (9)
C3—C2	1.540 (2)	O15—K20 ⁱ	2.777 (8)
C3—C4	1.541 (2)	O16—C6	1.263 (4)
C3—C6	1.5460 (19)	O16—Na19	2.537 (10)
C3—O17	1.427 (4)	O16—Na19 ^v	2.508 (8)
C4—C3	1.541 (2)	O16—K20 ^{vi}	2.660 (7)
C4—C5	1.511 (2)	O17—C3	1.427 (4)
C5—C4	1.511 (2)	O17—K20 ⁱⁱⁱ	2.717 (8)
C5—O13	1.286 (4)	Na19—O11	2.390 (10)
C5—O14	1.282 (4)	Na19—O12 ⁱ	3.138 (10)

C6—C3	1.5460 (19)	Na19—O14 ^{vii}	2.510 (9)
C6—O15	1.281 (4)	Na19—O15 ^{vii}	2.388 (9)
C6—O16	1.263 (4)	Na19—O15 ^v	2.512 (9)
O11—C1	1.260 (4)	Na19—O16	2.537 (10)
O11—Na19	2.390 (10)	Na19—O16 ^v	2.508 (8)
O11—K20 ⁱ	3.591 (9)	K20—O11 ⁱ	3.591 (9)
O12—C1	1.268 (4)	K20—O12 ^{vii}	3.159 (9)
O12—Na19 ⁱ	3.138 (10)	K20—O12	3.100 (8)
O12—K20	3.100 (8)	K20—O13 ⁱⁱⁱ	2.646 (8)
O12—K20 ⁱⁱ	3.159 (9)	K20—O14 ^{viii}	2.737 (8)
O13—C5	1.286 (4)	K20—O15 ⁱ	2.777 (8)
O13—K20 ⁱⁱⁱ	2.646 (8)	K20—O16 ^{ix}	2.660 (7)
O14—C5	1.282 (4)	K20—O17 ^{vii}	2.717 (8)
C2—C1—O11	115.5 (7)	C3—O17—K20 ⁱⁱ	137.1 (5)
C2—C1—O12	119.5 (6)	O11—Na19—O14 ^{vii}	101.9 (3)
O11—C1—O12	119.9 (5)	O11—Na19—O15 ^{vii}	97.2 (3)
C1—C2—C3	111.3 (4)	O11—Na19—O15 ^v	165.6 (4)
C2—C3—C4	106.1 (4)	O11—Na19—O16	84.1 (3)
C2—C3—C6	109.6 (4)	O11—Na19—O16 ^v	113.5 (4)
C2—C3—O17	110.5 (4)	O14 ^{vii} —Na19—O15 ^{vii}	78.7 (3)
C4—C3—C6	109.8 (4)	O14 ^{vii} —Na19—O15 ^v	88.4 (3)
C4—C3—O17	112.8 (4)	O14 ^{vii} —Na19—O16	77.0 (3)
C6—C3—O17	108.0 (4)	O14 ^{vii} —Na19—O16 ^v	133.6 (3)
C3—C4—C5	115.8 (5)	O15 ^{vii} —Na19—O15 ^v	94.7 (3)
C4—C5—O13	116.5 (4)	O15 ^{vii} —Na19—O16	155.4 (4)
C4—C5—O14	122.2 (6)	O15 ^{vii} —Na19—O16 ^v	123.0 (4)
O13—C5—O14	121.2 (6)	O15 ^v —Na19—O16	88.5 (3)
C3—C6—O15	118.8 (4)	O15 ^v —Na19—O16 ^v	52.60 (18)
C3—C6—O16	118.9 (4)	O16—Na19—O16 ^v	77.9 (3)
O15—C6—O16	121.9 (5)	O12 ^{vii} —K20—O12	146.9 (3)
C1—O11—Na19	134.1 (7)	O12 ^{vii} —K20—O13 ⁱⁱⁱ	91.1 (2)
C1—O12—K20	115.3 (5)	O12 ^{vii} —K20—O14 ^{viii}	71.8 (2)
C1—O12—K20 ⁱⁱ	97.7 (5)	O12 ^{vii} —K20—O15 ⁱ	68.5 (2)
K20—O12—K20 ⁱⁱⁱ	146.9 (3)	O12 ^{vii} —K20—O16 ^{ix}	135.5 (3)
C5—O13—K20 ⁱⁱⁱ	142.9 (5)	O12 ^{vii} —K20—O17 ^{vii}	71.9 (2)
C5—O14—Na19 ⁱⁱ	151.2 (7)	O12—K20—O13 ⁱⁱⁱ	94.6 (2)
C5—O14—K20 ^{iv}	125.9 (6)	O12—K20—O14 ^{viii}	140.9 (3)
Na19 ⁱⁱ —O14—K20 ^{iv}	82.7 (2)	O12—K20—O15 ⁱ	115.0 (3)
C6—O15—Na19 ⁱⁱ	115.3 (8)	O12—K20—O16 ^{ix}	75.2 (2)
C6—O15—Na19 ^v	89.5 (4)	O12—K20—O17 ^{vii}	75.6 (2)
C6—O15—K20 ⁱ	125.5 (7)	O13 ⁱⁱⁱ —K20—O14 ^{viii}	73.0 (2)
Na19 ⁱⁱ —O15—Na19 ^v	85.3 (3)	O13 ⁱⁱⁱ —K20—O15 ⁱ	149.4 (2)
Na19 ⁱⁱ —O15—K20 ⁱ	117.4 (3)	O13 ⁱⁱⁱ —K20—O16 ^{ix}	100.9 (3)
Na19 ^v —O15—K20 ⁱ	81.9 (3)	O13 ⁱⁱⁱ —K20—O17 ^{vii}	89.3 (2)
C6—O16—Na19	110.5 (7)	O14 ^{viii} —K20—O15 ⁱ	78.9 (2)
C6—O16—Na19 ^v	90.1 (4)	O14 ^{viii} —K20—O16 ^{ix}	71.2 (3)
C6—O16—K20 ^{vi}	164.8 (7)	O14 ^{viii} —K20—O17 ^{vii}	139.0 (3)

Na19—O16—Na19 ^v	102.2 (3)	O15 ⁱ —K20—O16 ^{ix}	80.8 (2)
Na19—O16—K20 ^{vi}	83.8 (3)	O15 ⁱ —K20—O17 ^{vii}	104.6 (3)
Na19 ^v —O16—K20 ^{vi}	92.0 (3)	O16 ^{ix} —K20—O17 ^{vii}	149.7 (3)
C3—O17—H18	108.0 (4)		

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

(ramm093_DFT)*Crystal data*

NaKHC ₆ H ₅ O ₇	$c = 10.1419 \text{ \AA}$
$M_r = 252.17$	$\alpha = 74.8964^\circ$
Triclinic, $P1$	$\beta = 76.0187^\circ$
Hall symbol: $-P 1$	$\gamma = 71.4496^\circ$
$a = 5.9993 \text{ \AA}$	$V = 451.27 \text{ \AA}^3$
$b = 8.2277 \text{ \AA}$	$Z = 2$

Data collection

Density functional calculation

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	0.58473	0.47461	0.69008	0.02150*
C2	0.58840	0.56265	0.80315	0.00260*
C3	0.77491	0.66691	0.77024	0.00260*
C4	0.74288	0.74284	0.89935	0.00260*
C5	0.90458	0.85546	0.89044	0.02150*
C6	0.74020	0.81902	0.64154	0.02150*
H7	0.62719	0.46044	0.89439	0.00340*
H8	0.41202	0.65122	0.82730	0.00340*
H9	0.77282	0.63557	0.98912	0.00340*
H10	0.55890	0.82308	0.92211	0.00340*
O11	0.49077	0.58291	0.58433	0.02150*
O12	0.65591	0.31455	0.69939	0.02150*
O13	0.88392	0.90257	1.00623	0.02150*
O14	1.04072	0.89738	0.78176	0.02150*
O15	0.90892	0.81600	0.53821	0.02150*
O16	0.54704	0.93850	0.64884	0.02150*
O17	1.00409	0.54419	0.74830	0.02150*
H18	1.10980	0.60623	0.68105	0.02790*
Na19	0.26024	0.87640	0.55043	0.05110*
K20	0.17511	0.20585	0.71480	0.04060*
H21	0.50000	0.50000	0.50000	0.03000*
H22	1.00000	1.00000	1.00000	0.03000*

Bond lengths (\AA)

C1—C2	1.515	C4—H10	1.095
C1—O11	1.318	C5—O13	1.297

C1—O12	1.234	C5—O14	1.244
C2—C3	1.543	C6—O15	1.268
C2—H7	1.092	C6—O16	1.259
C2—H8	1.090	O11—H21	1.207
C3—C4	1.540	O13—H22	1.200
C3—C6	1.558	O17—H18	0.971
C3—O17	1.430	H21—O11 ⁱ	1.207
C4—C5	1.515	H22—O13 ⁱⁱ	1.200
C4—H9	1.095		

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+2, -y+2, -z+2$.

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O13—H22...O13	1.200	1.200	2.400	180.0
O11—H21...O11	1.207	1.207	2.414	180.0
O17—H18...O15	0.971	2.179	2.676	110.3
O17—H18...O11	0.971	2.227	3.060	143.1