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A second polymorph of sodium dihydrogen citrate, $NaH_2C_6H_5O_7$: structure solution from powder diffraction data and DFT comparison

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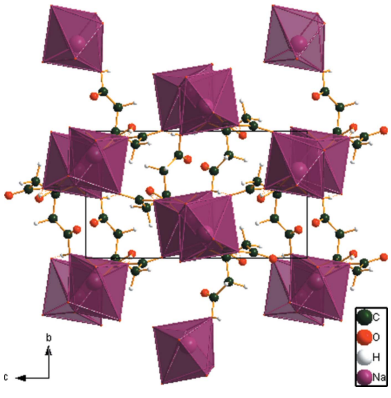
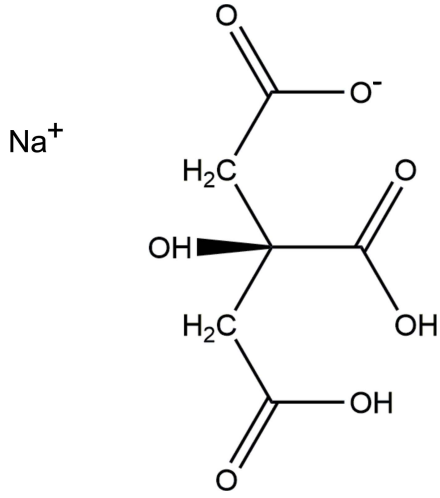
The crystal structure of a second polymorph of sodium dihydrogen citrate, $Na^+\cdot H_2C_6H_5O_7^-$, has been solved and refined using laboratory X-ray powder diffraction data, and optimized using density functional techniques. The powder pattern of the commercial sample used in this study did not match that corresponding to the known crystal structure [Glusker *et al.* (1965). *Acta Cryst.* **19**, 561–572; refcode NAHCIT]. In this polymorph, the [NaO_7] coordination polyhedra form edge-sharing chains propagating along the *a* axis, while in NAHCIT the octahedral [NaO_6] groups form edge-sharing pairs bridged by two hydroxy groups. The most notable difference is that in this polymorph one of the terminal carboxyl groups is deprotonated, while in NAHCIT the central carboxylate group is deprotonated, as is more typical.

1. Chemical context

In the course of a systematic study of the crystal structures of Group 1 (alkali metal) citrate salts to better understand the anion's conformational flexibility, deprotonation mode, coordination tendencies, and hydrogen bonding, we have determined several new crystal structures. Most of the new structures were solved using powder diffraction data (laboratory and/or synchrotron), but single crystals were used where available. The general trends and conclusions about the 16 new compounds and 12 previously characterized structures are being reported separately (Rammohan & Kaduk, 2016*a*). Three of the new structures – $NaKHC_6H_5O_7$, $NaK_2C_6H_5O_7$, and $Na_3C_6H_5O_7$ – have been published recently (Rammohan & Kaduk, 2016*b*,*c*,*d*).





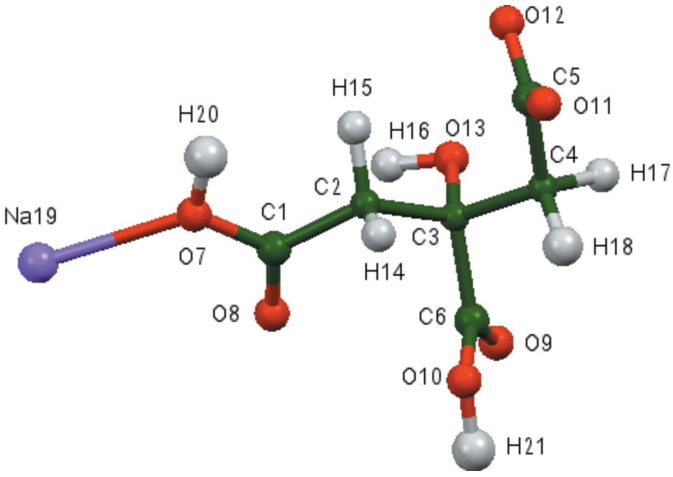


Figure 1
The asymmetric unit, showing the atom numbering. The atoms are represented by 50% probability spheroids.

2. Structural commentary

The asymmetric unit of the title compound is shown in Fig. 1. The root-mean-square deviation of the non-hydrogen atoms in the Rietveld-refined and DFT-optimized structures is 0.148 Å. The maximum deviation is 0.318 Å, at the sodium ion. The good agreement between the two structures (Fig. 2) is strong evidence that the experimental structure is correct (van de Streek & Neumann, 2014). This discussion uses the DFT-optimized structure. All of the bond lengths, bond angles, and most torsion angles fall within the normal ranges indicated by a *Mercury Mogul* geometry check (Macrae *et al.*, 2008). Only the C2—C3—C4—C5 torsion angle is flagged as unusual. It lies in the tail of a minority *gauche* population of similar torsion angles. The citrate anion occurs in the *gauche,trans*-conformation, which is one of the two low-energy conformations of an isolated citrate ion. The central carboxylate group and the hydroxy group occur in the normal planar arrangement. The citrate chelates to one Na19 ion through the central carboxyl O9 atom and the hydroxy group O13, and to a second Na19 ion through the terminal carboxyl atom O12 and the hydroxy group O13. The Na^+ ion is seven-coordinate (pentagonal–bipyramidal), and has a bond-valence sum of 1.12.

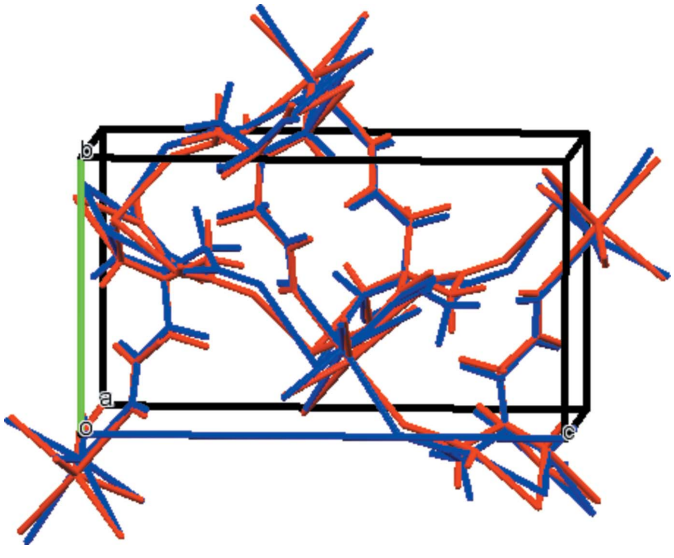


Figure 2
Comparison of the refined and optimized structures of sodium dihydrogen citrate. The refined structure is in red, and the DFT-optimized structure is in blue.

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H20···O11	1.01	1.61	2.627	176
O10—H21···O12	1.04	1.46	2.498	175
O13—H16···O8	0.97	2.50	3.033	114
C2—H15···O8	1.09	2.50	3.166	119

3. Supramolecular features

In this polymorph, the [NaO_7] coordination polyhedra (Fig. 3) form edge-sharing chains propagating along the *a* axis, while in NAHCIT (Glusker *et al.*, 1965), the octahedral [NaO_6] units form edge-sharing pairs bridged by two hydroxy groups.

The conformations of the citrate ions in the two structures are similar. The root-mean-square displacement of the non-hydrogen atoms is 0.11 Å. The conformations of the hydroxy groups differ, reflecting differences in coordination and hydrogen bonding. The most notable difference is that in this polymorph, one of the terminal carboxyl groups is deprotonated, while in NAHCIT the central carboxylate group is deprotonated, as is more typical.

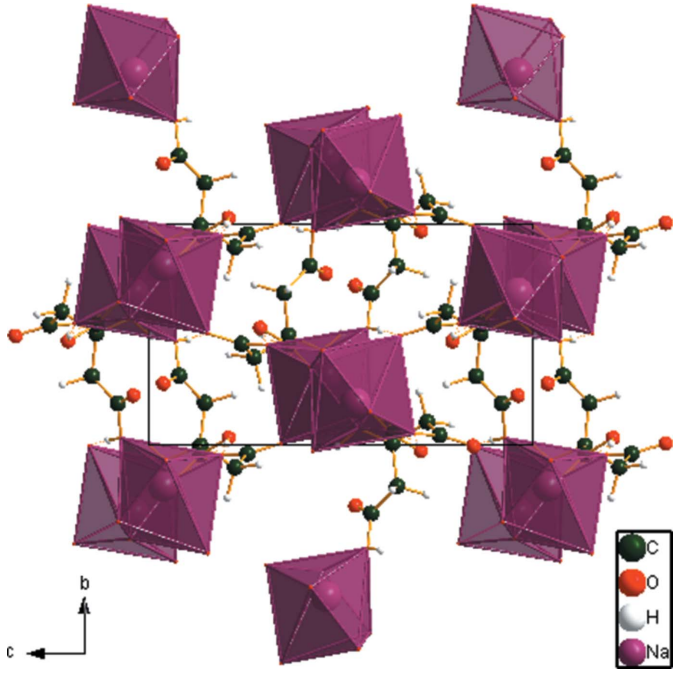


Figure 3
Crystal structure of $NaH_2C_6H_5O_7$, viewed down the *a* axis.

Table 2
Experimental details.

	Phase 1	Phase 2
Crystal data		
Chemical formula	$Na^+ \cdot C_6H_7O_7^-$	Si
M_r	214.10	28.09
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Cubic, $Fd\bar{3}m$
Temperature (K)	300	300
a, b, c (Å)	7.4527 (3), 7.7032 (3), 13.4551 (4)	5.43105, 5.43105, 5.43105
α, β, γ (°)	90, 90, 90	90, 90, 90
V (Å^3)	772.45 (5)	160.20
Z	4	8
Radiation type	$K\alpha_1$, $K\alpha_2$, λ = 1.540629, 1.544451 Å	$K\alpha_1$, $K\alpha_2$, λ = 1.540629, 1.544451 Å
Specimen shape, size (mm)	Flat sheet, 25 × 25	Flat sheet, 25 × 25
Data collection		
Diffractometer	Bruker D2 Phaser	Bruker D2 Phaser
Specimen mounting	Bruker PMMA holder	Bruker PMMA holder
Data collection mode	Reflection	Reflection
Scan method	Step	Step
2θ values (°)	$2\theta_{min}$ = 5.042 $2\theta_{max}$ = 100.048 $2\theta_{step}$ = 0.020	$2\theta_{min}$ = 5.042 $2\theta_{max}$ = 100.048 $2\theta_{step}$ = 0.020
Refinement		
R factors and goodness of fit	R_p = 0.063, R_{wp} = 0.084, R_{exp} = 0.024, $R(F^2)$ = 0.0780, χ^2 = 12.180	R_p = 0.063, R_{wp} = 0.084, R_{exp} = 0.024, $R(F^2)$ = 0.0780, χ^2 = 12.180
No. of parameters	76	76
No. of restraints	29	29

The same symmetry and lattice parameters were used for the DFT calculation. Computer programs: *DIFFRAC.Measurement* (Bruker, 2009), *Powder4* (Dragoe, 2001), *DASH* (David *et al.*, 2006), *GSAS* (Larson & Von Dreele, 2004), *EXPGUI* (Toby, 2001), *DIAMOND* (Crystal Impact, 2015) and *publCIF* (Westrip, 2010).

In this form, the hydrogen bonds occur in layers in the *ab* plane, while in NAHCIT the hydrogen bonds form double-ladder chains along the *c* axis. The hydrogen bonds in this form contribute about 4.3 kcal mol^{-1} more to the lattice energy than those in NAHCIT, and seem to include a C—H···O hydrogen bond (Table 1). Comparison of the DFT energies of the two polymorphs shows that this polymorph is 3.24 kcal mol^{-1} higher in energy than NAHCIT. Presumably it was crystallized at a higher temperature than NAHCIT, which was crystallized at 343 K.

4. Database survey

Details of the comprehensive literature search for citrate structures are presented in Rammohan & Kaduk (2016*a*). The crystal structure of sodium dihydrogen citrate is reported in Glusker *et al.* (1965), and the powder pattern calculated from this structure is PDF entry 02-063-5032. The observed powder pattern matched PDF entry 00-016-1182 (de Wolff *et al.*, 1966) A reduced cell search of the cell of the observed polymorph in the Cambridge Structural Database (Groom *et al.*, 2016) (increasing the default tolerance from 1.5 to 2.0%) yielded 223 hits, but limiting the chemistry to C, H, Na, and O only resulted in no hits. The powder pattern is now contained in the the Powder Diffraction File (ICDD, 2015) as entry 00-063-1340.

5. Synthesis and crystallization

The sample was purchased from Sigma–Aldrich (lot #BCBC0142). Before measuring the powder pattern, a portion of the sample was ground in a Spex 8000 mixer/mill and blended with a NIST SRM 640b silicon internal standard.

6. Refinement details

The powder pattern was indexed using DICVOL06 (Louër & Boultif, 2007). The background and $K\alpha_2$ peaks were removed using *Jade* (MDI, 2012), and *Powder4* (Dragoe, 2001) was used to convert the data into an XYE file. The 10–52.22° portion of the pattern was processed in *DASH 3.2* (David *et al.*, 2006), which suggested $P2_12_12_1$ as the most probable space group. Citrate and Na fragments were used to solve the structure in this space group using *DASH*.

The powder pattern (Fig. 4) was indexed using *Jade 9.5* (MDI, 2012). Pseudo-Voigt profile coefficients were as parameterized in Thompson *et al.* (1987) with profile coefficients for Simpson's rule integration of the Pseudo-Voigt function according to Howard (1982). The asymmetry correction of Finger *et al.* (1994) was applied and microstrain broadening by Stephens (1999).

The structure was refined by the Rietveld method using *GSAS/EXPGUI* (Larson & Von Dreele, 2004: Toby, 2001). All C—C and C—O bond lengths were restrained, as were all bond angles. The hydrogen atoms were included at fixed positions, which were recalculated during the course of the refinement using *Materials Studio* (Dassault Systemes, 2014). The U_{iso} values of the atoms in the central and outer portions of the citrate 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.

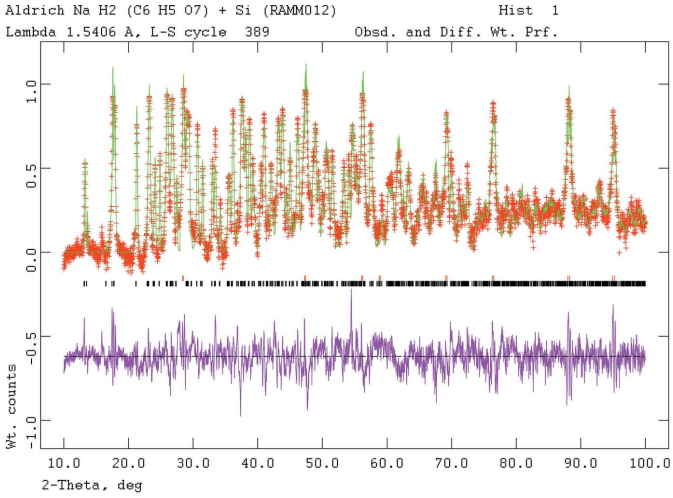


Figure 4
Rietveld plot for the refinement of $NaH_2C_6H_5O_7$. The vertical scale is not the raw counts but the counts multiplied by the least-squares weights. This plot emphasizes the fit of the weaker peaks. The red crosses represent the observed data points, and the green line is the calculated pattern. The magenta curve is the difference pattern, plotted at the same scale as the other patterns. The 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.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) morphology suggests that we might expect a blocky morphology for this phase. A 4th-order spherical harmonic texture model was included in the refinement. The texture index was 1.374, indicating that preferred orientation was significant for this rotated-flat-plate specimen.

7. DFT calculations

Crystal data, data collection and structure refinement details are summarized in Table 2. 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 Na was that of Dovesi *et al.* (1991). The calculation used 8 *k*-points and the B3LYP functional, and took about 60 h on a 2.4 GHz PC. The U_{iso} from the Rietveld were assigned to the optimized fractional coordinates.

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supporting information

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A second polymorph of sodium dihydrogen citrate, $NaH_2C_6H_5O_7$: structure solution from powder diffraction data and DFT comparison

Alagappa Rammohan and James A. Kaduk

Computing details

(RAMM012A_phase_1) Sodium dihydrogen citrate

Crystal data

$Na^+ \cdot C_6H_7O_7^-$
M_r = 214.10
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 7.4527 (3) Å
b = 7.7032 (3) Å
c = 13.4551 (4) Å
V = 772.45 (5) Å^3
Z = 4
D_x = 1.841 Mg m^{-3}
T = 300 K

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

	x	y	z	U_{iso}*/U_{eq}
Na1	0.8787 (12)	−0.2363 (10)	−0.0501 (5)	0.035 (2)*
C2	0.870 (2)	0.1658 (19)	0.0733 (10)	0.0258 (15)*
C3	0.769 (2)	0.282 (2)	0.1478 (9)	0.019 (3)*
C4	0.804 (2)	0.4793 (18)	0.1383 (8)	0.019 (3)*
C5	0.723 (2)	0.564 (2)	0.2299 (10)	0.019 (3)*
C6	0.528 (3)	0.509 (2)	0.2460 (9)	0.0258 (15)*
C7	1.006 (3)	0.528 (2)	0.1354 (9)	0.0258 (15)*
O8	0.8117 (18)	0.0107 (14)	0.0697 (7)	0.0258 (15)*
O9	1.0081 (19)	0.2131 (15)	0.0280 (6)	0.0258 (15)*
O10	0.5110 (16)	0.4578 (14)	0.3329 (6)	0.0258 (15)*
O11	0.418 (2)	0.5412 (14)	0.1838 (7)	0.0258 (15)*
O12	1.062 (2)	0.6521 (14)	0.0763 (6)	0.0258 (15)*
O13	1.081 (2)	0.4474 (15)	0.2023 (7)	0.0258 (15)*
O14	0.7172 (16)	0.5561 (13)	0.0514 (6)	0.0258 (15)*
H15	0.74818	0.24337	0.22455	0.025 (4)*
H16	0.61496	0.2687	0.13343	0.025 (4)*
H17	0.69716	0.46671	0.01345	0.034 (2)*
H18	0.70618	0.69511	0.23029	0.025 (4)*
H19	0.76628	0.53492	0.29702	0.025 (4)*
H20	0.7027	−0.0047	0.0991	0.039*
H21	0.2904	0.5783	0.1915	0.039*

Geometric parameters (Å, º)

Na1—O8	2.543 (12)	C7—O12	1.314 (13)
Na1—O10^{i}	2.463 (13)	C7—O13	1.227 (13)
Na1—O11^{ii}	2.363 (13)	O8—Na1	2.543 (12)
Na1—O12^{iii}	2.344 (13)	O8—C2	1.271 (14)
Na1—O12^{iv}	2.475 (16)	O8—H20	0.912 (13)
Na1—O14^{iii}	2.423 (13)	O9—C2	1.253 (14)
Na1—O14^{ii}	2.879 (13)	O10—Na1^{v}	2.463 (13)
C2—C3	1.540 (10)	O10—C6	1.240 (12)
C2—O8	1.271 (14)	O11—Na1^{iv}	2.363 (13)
C2—O9	1.253 (14)	O11—C6	1.198 (14)
C3—C2	1.540 (10)	O11—H21	0.998 (15)
C3—C4	1.546 (10)	O12—Na1^{vi}	2.344 (13)
C3—H15	1.087 (12)	O12—Na1^{ii}	2.475 (16)
C3—H16	1.167 (18)	O12—C7	1.314 (13)
C4—C3	1.546 (10)	O13—C7	1.227 (13)
C4—C5	1.519 (10)	O14—Na1^{vi}	2.423 (13)
C4—C7	1.549 (10)	O14—Na1^{iv}	2.879 (13)
C4—O14	1.461 (9)	O14—C4	1.461 (9)
C5—C4	1.519 (10)	O14—H17	0.871 (10)
C5—C6	1.528 (10)	H15—C3	1.087 (12)
C5—H18	1.022 (16)	H16—C3	1.167 (18)
C5—H19	0.984 (15)	H17—O14	0.871 (10)
C6—C5	1.528 (10)	H18—C5	1.022 (16)
C6—O10	1.240 (12)	H19—C5	0.984 (15)
C6—O11	1.198 (14)	H20—O8	0.912 (13)
C7—C4	1.549 (10)	H21—O11	0.998 (15)
O8—Na1—O10^{i}	171.5 (5)	C3—C4—C7	113.8 (12)
O8—Na1—O11^{ii}	91.8 (4)	C3—C4—O14	112.9 (11)
O8—Na1—O12^{iii}	85.9 (5)	C5—C4—C7	107.7 (13)
O8—Na1—O12^{iv}	73.0 (5)	C5—C4—O14	107.5 (12)
O8—Na1—O14^{iii}	92.2 (4)	C7—C4—O14	108.2 (12)
O8—Na1—H21^{ii}	94.0 (4)	C4—C5—C6	112.1 (16)
O10^{i}—Na1—O11^{ii}	85.0 (4)	C5—C6—O10	108.5 (13)
O10^{i}—Na1—O12^{iii}	90.9 (4)	C5—C6—O11	119.6 (15)
O10^{i}—Na1—O12^{iv}	114.1 (4)	O10—C6—O11	131.0 (19)
O10^{i}—Na1—O14^{iii}	94.0 (4)	C4—C7—O12	120.0 (15)
O11^{ii}—Na1—O12^{iii}	135.6 (6)	C4—C7—O13	107.6 (13)
O11^{ii}—Na1—O12^{iv}	81.0 (5)	O12—C7—O13	131.8 (17)
O11^{ii}—Na1—O14^{iii}	155.6 (5)	Na1—O8—C2	131.4 (11)
O12^{iii}—Na1—O12^{iv}	139.0 (5)	Na1^{v}—O10—C6	142.0 (14)
O12^{iii}—Na1—O14^{iii}	68.8 (4)	Na1^{iv}—O11—C6	138.6 (12)
O12^{iv}—Na1—O14^{iii}	77.2 (4)	Na1^{vi}—O12—Na1^{ii}	110.8 (4)
C3—C2—O8	114.0 (13)	Na1^{vi}—O12—C7	121.4 (12)
C3—C2—O9	123.3 (15)	Na1^{ii}—O12—C7	125.6 (12)
O8—C2—O9	122.3 (15)	Na1^{vi}—O14—C4	119.9 (10)

C2—C3—C4	115.8 (13)	Na1^{iv}—H21—O11	78.3 (6)
C3—C4—C5	106.5 (13)		

Symmetry codes: (i) −*x*+3/2, −*y*, *z*−1/2; (ii) *x*+1/2, −*y*+1/2, −*z*; (iii) *x*, *y*−1, *z*; (iv) *x*−1/2, −*y*+1/2, −*z*; (v) −*x*+3/2, −*y*, *z*+1/2; (vi) *x*, *y*+1, *z*.

(RAMM012A_phase_2)

Crystal data

Si	a = 5.43105 Å
M_r = 28.09	V = 160.20 Å^3
Cubic, $Fd\bar{3}m$	Z = 8
Hall symbol: -F 4vw 2vw	T = 300 K

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	0.0304 (5)*

Geometric parameters (Å, º)

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

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

(ramm012a_DFT)

Crystal data

$C_6H_7NaO_7$	c = 13.4551 Å
M_r = 214.10	V = 772.45 Å^3
Orthorhombic, $P2_12_12_1$	Z = 4
a = 7.4527 Å	None; DFT calculation radiation
b = 7.7032 Å	T = 300 K

Data collection

h = →	l = →
k = →	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.86930	0.18689	0.07976	0.02580*
C2	0.76864	0.31082	0.14718	0.01910*
C3	0.80887	0.50489	0.13526	0.01910*
C4	0.72870	0.60356	0.22535	0.01910*
C5	0.54276	0.54256	0.25725	0.02580*
C6	1.01121	0.54559	0.13579	0.02580*
O7	0.79690	0.02935	0.07081	0.02580*

O8	1.01065	0.22287	0.03877	0.02580*
O9	1.07983	0.64886	0.07729	0.02580*
O10	1.09698	0.46975	0.20891	0.02580*
O11	0.51997	0.49361	0.34617	0.02580*
O12	0.42047	0.54898	0.19138	0.02580*
O13	0.73153	0.57264	0.04623	0.02580*
H14	0.79934	0.27249	0.22351	0.02500*
H15	0.62504	0.29365	0.13548	0.02500*
H16	0.75707	0.49090	−0.00712	0.02500*
H17	0.71973	0.74075	0.20546	0.02500*
H18	0.81890	0.59174	0.28862	0.03350*
Na19	0.91363	−0.18930	−0.03840	0.03460*
H20	0.67516	0.02054	0.10415	0.03900*
H21	0.23334	0.49718	0.20429	0.03900*

Bond lengths (Å)

C1—C2	1.516	C4—H17	1.092
C1—O7	1.334	C4—H18	1.089
C1—O8	1.221	C5—O11	1.266
C2—C3	1.533	C5—O12	1.272
C2—H14	1.093	C6—O9	1.230
C2—H15	1.090	C6—O10	1.311
C3—C4	1.550	O7—H20	1.014
C3—C6	1.540	O10—H21^{i}	1.040
C3—O13	1.428	O13—H16	0.974
C4—C5	1.525	H21—O10^{ii}	1.040

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H20···O11	1.01	1.61	2.627	176
O10—H21···O12	1.04	1.46	2.498	175
O13—H16···O8	0.97	2.50	3.033	114
C2—H15···O8	1.09	2.50	3.166	119