

Crystal structure of pentasodium hydrogen dicitrate from synchrotron X-ray powder diffraction data and DFT comparison

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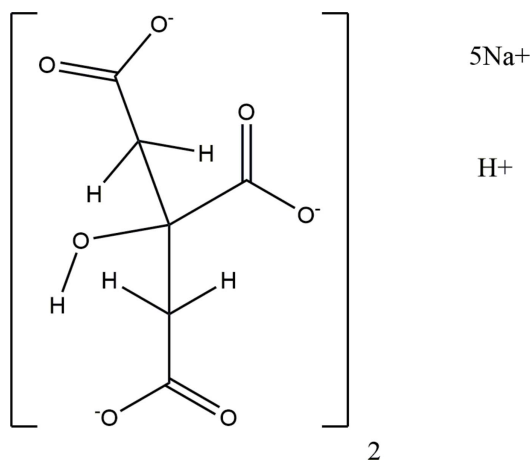
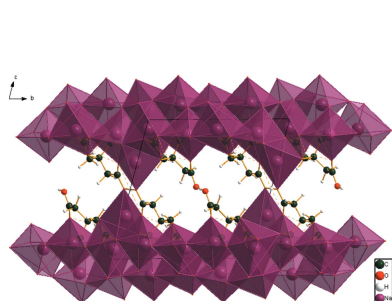
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The crystal structure of pentasodium hydrogen dicitrate, $\text{Na}_5\text{H}(\text{C}_6\text{H}_5\text{O}_7)_2$, has been solved and refined using synchrotron X-ray powder diffraction data, and optimized using density functional techniques. Each of the two independent citrate anions is joined into a dimer by very strong centrosymmetric $\text{O} \cdots \text{O}$ hydrogen bonds, with $\text{O} \cdots \text{O}$ distances of 2.419 and 2.409 Å. Four octahedrally coordinated Na^+ ions share edges to form open layers parallel to the *ab* plane. A fifth Na^+ ion in trigonal-bipyramidal coordination shares faces with NaO_6 octahedra on both sides of these layers.

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 X-ray powder diffraction data (laboratory and/or synchrotron), but single crystals were used where available. The general trends and conclusions about the sixteen new compounds and twelve previously characterized structures are being reported separately (Rammohan & Kaduk, 2017*a*). Nine of the new structures – $\text{NaKHC}_6\text{H}_5\text{O}_7$, $\text{NaK}_2\text{C}_6\text{H}_5\text{O}_7$, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$, $\text{NaH}_2\text{C}_6\text{H}_5\text{O}_7$, $\text{Na}_2\text{HC}_6\text{H}_5\text{O}_7$, $\text{K}_3\text{C}_6\text{H}_5\text{O}_7$, $\text{Rb}_2\text{HC}_6\text{H}_5\text{O}_7$, $\text{Rb}_3\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})$, and $\text{Rb}_3\text{C}_6\text{H}_5\text{O}_7$ – have been published recently (Rammohan & Kaduk, 2016*a,b,c,d,e*, 2017*b,c,d*; Rammohan *et al.*, 2016), and two additional structures – $\text{KH}_2\text{C}_6\text{H}_5\text{O}_7$ and $\text{KH}_2\text{C}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})_2$ – have been communicated to the CSD (Kaduk & Stern, 2016*a,b*).



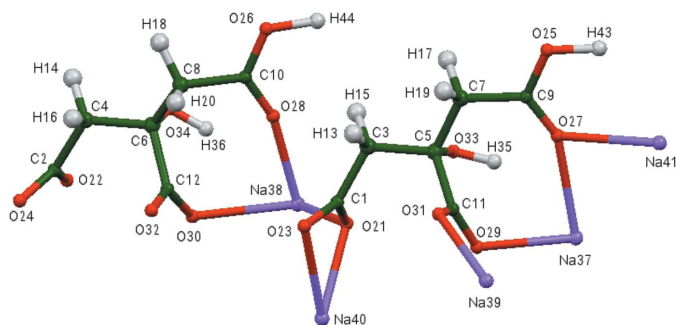


Figure 1

The asymmetric unit of $\text{Na}_5\text{H}(\text{C}_6\text{H}_5\text{O}_7)_2$, with the atom numbering. The atoms are represented by 50% probability spheroids.

2. Structural commentary

The compound $\text{Na}_5\text{H}(\text{C}_6\text{H}_5\text{O}_7)_2$ was unexpectedly synthesized by heating $\text{Na}_2\text{HC}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})_{1.5}$. 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 0.216 Å (Fig. 2). The reasonable agreement between the two structures is 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). Both the O28—C10 bond length of 1.249 Å [Z-score = 3.4; average = 1.213 (13) Å] and the O28—C10—C8 angle of 120.4° [Z-score = 4.0; average = 126.9 (16)°] are flagged as unusual. Since this oxygen atom also coordinates to an Na^+ cation, it is not unreasonable to encounter some slightly unusual geometry. Each Na^+ cation is chelated by at least one citrate anion. The chelation modes include hydroxyl/terminal carboxyl, terminal/central carboxyl, and coordination of both oxygen atoms of a terminal carboxyl group.

The structure contains five independent Na^+ cations. Na37, Na38, Na39, and Na40 exhibit octahedral coordination spheres, with bond-valence sums of 1.19, 1.24, 1.04, and 1.17

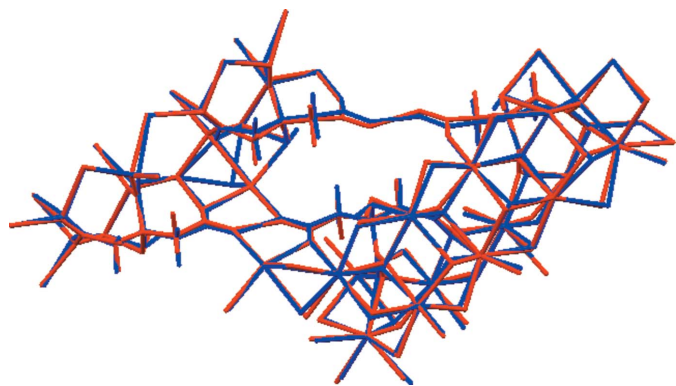


Figure 2

Comparison of the refined and optimized structures of pentasodium hydrogen dicitrate. The refined structure is in red, and the DFT-optimized structure is in blue.

Table 1

Hydrogen-bond geometry (Å, °) for $\text{Na}_5\text{Hcit}_2\text{-DFT}$.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O26—H44 \cdots O26 ⁱ	1.210	1.210	2.419	180.0
O25—H43 \cdots O25 ⁱⁱ	1.205	1.205	2.409	180.0
O33—H35 \cdots O23 ⁱⁱⁱ	0.975	2.045	2.799	132.8
O33—H35 \cdots O29	0.975	2.450	2.664	91.7
O34—H36 \cdots O30	0.971	2.212	2.631	104.6
O34—H36 \cdots O28	0.971	2.302	2.831	113.4
O34—H36 \cdots O24 ⁱⁱⁱ	0.971	2.385	3.223	144.3

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

valence units, respectively. Na41 is only five-coordinate with a trigonal-bipyramidal coordination sphere, but its bond-valence sum is 1.26. The only O atom not coordinating to an Na^+ cation is O26, which participates in very strong centrosymmetric hydrogen bonds. The octahedra involving Na37—Na40 share edges to form open layers parallel to the *ab* plane. Trigonal-bipyramidal Na41O_5 polyhedra share faces with Na37O_6 and Na39O_6 octahedra on both sides of these layers. The crystal structure is illustrated in Fig. 3.

The Mulliken overlap populations and atomic charges indicate that the metal–oxygen bonding is ionic. Comparison of the structures of the starting material $\text{Na}_2\text{HC}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})_{1.5}$ and the title compound does not suggest any plausible mechanism for the conversion.

The Bravais–Friedel–Donnay–Harker (Bravais, 1866; Friedel, 1907; Donnay & Harker, 1937) morphology suggests that we might expect elongated morphology for pentasodium hydrogen dicitrate, with [100] as the principal axis. A 4th-order spherical harmonic texture model was included in the refinement. The texture index was 1.014, indicating that preferred orientation was slight for this rotated capillary specimen.

3. Supramolecular features

The layers are connected by very strong centrosymmetric O26—H44 \cdots O26 and O25—H43 \cdots O25 hydrogen bonds (Table 1). The O26 \cdots O26 distance is 2.419 Å and the

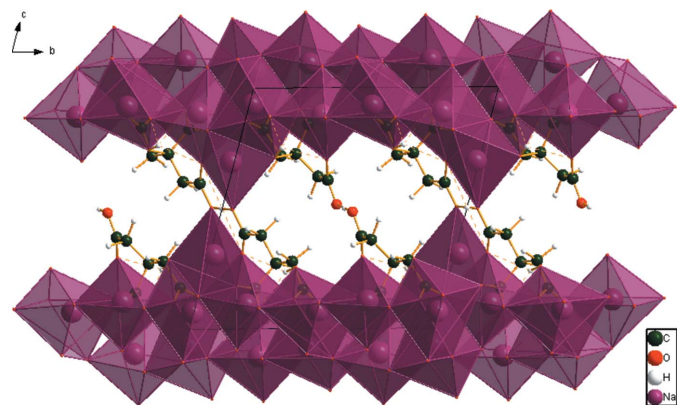


Figure 3

Crystal structure of $\text{Na}_5\text{H}(\text{C}_6\text{H}_5\text{O}_7)_2$, viewed down the *a* axis.

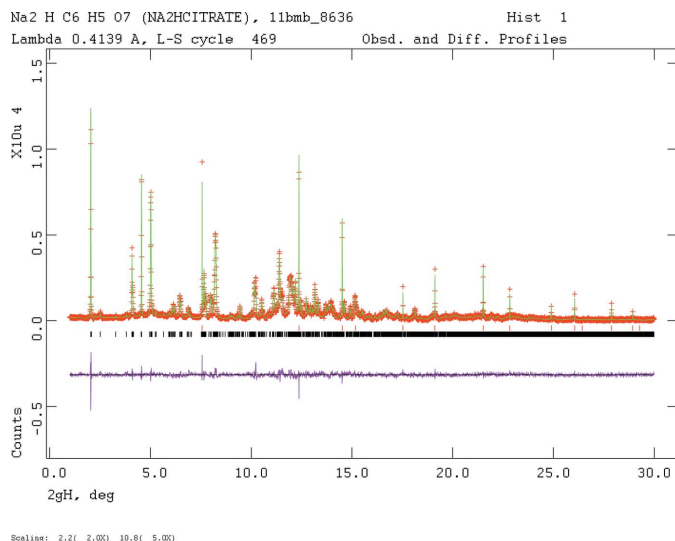


Figure 4

Rietveld plot for the refinement of $\text{Na}_5\text{H}(\text{C}_6\text{H}_5\text{O}_7)_2$. 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 2.0 at 2.2° , and by 5.0 at 10.8° . The row of black tick marks indicates the reflection positions. The red tick marks indicate the positions of the peaks of the Si internal standard.

$\text{O}25 \cdots \text{O}25$ distance is 2.409 \AA , making these among the shortest hydrogen bonds. The Mulliken overlap populations in the hydrogen bonds are 0.145 and 0.136 e , which correspond to 20.8 and 20.2 kcal mol^{-1} for each hydrogen bond (Rammohan & Kaduk, 2017a). These hydrogen bonds link two citrates into dimers.

The Mulliken overlap populations indicate that the hydroxyl groups $\text{O}33\text{—H}35$ and $\text{O}34\text{—H}36$ each act as donors in three hydrogen bonds. One [with graph set $S(5)$] is to the central carboxylate group, and another is intramolecular to a terminal carboxyl group. The third hydrogen bond is intermolecular. These hydrogen bonds are much weaker than the centrosymmetric ones, contributing 5–8 kcal mol^{-1} to the crystal energy.

4. Database survey

Details of the comprehensive literature search for citrate structures are presented in Rammohan & Kaduk (2017a). A reduced cell search of the cell of pentasodium hydrogen dicitrate in the Cambridge Structural Database (Groom *et al.*, 2016) (increasing the default tolerance from 1.5 to 2.0%) yielded 98 hits, but combining the cell search with the elements C, H, Na, and O only yielded no hits.

5. Synthesis and crystallization

The title compound was prepared by heating commercial reagent $\text{Na}_2\text{HC}_6\text{H}_5\text{O}_7(\text{H}_2\text{O})_{1.5}$ (Sigma–Aldrich lot BCBC6031V) from 295–453 K at 14 K min^{-1} in air. The crystal structure of this reagent is reported in Rammohan *et al.*

Table 2

Experimental details.

Crystal data	
Chemical formula	$\text{Na}_5\text{H}(\text{C}_6\text{H}_5\text{O}_7)_2$
M_r	494.16
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	295
a, b, c (Å)	6.35262 (9), 11.98628 (18), 12.16544 (16)
α, β, γ (°)	73.8374 (13), 80.8808 (15), 80.7103 (10)
V (Å ³)	871.72 (2)
Z	2
Radiation type	Synchrotron, $\lambda = 0.413891 \text{ \AA}$
Specimen shape, size (mm)	Cylinder, 1.5×1.5
Data collection	
Diffractometer	11-BM APS
Specimen mounting	Kapton capillary
Data collection mode	Transmission
Scan method	Step
T_{\min} , T_{\max}	1.000, 1.000
2θ values (°)	$2\theta_{\min} = 0.5$ $2\theta_{\max} = 50.0$ $2\theta_{\text{step}} = 0.001$
Refinement	
R factors and goodness of fit	$R_p = 0.083$, $R_{wp} = 0.097$, $R_{\text{exp}} = 0.073$, $R(F^2) = 0.085$, $\chi^2 = 1.823$
No. of parameters	145
No. of restraints	58

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

(2016). After holding at 453 K for two minutes, the sample began to discolour (turn yellow), and it was taken from the oven and sealed in a glass jar to cool.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. The sample was blended with NIST SRM 640b silicon internal standard in a Spex 8000 mixer/mill, and packed into a standard sample holder. It was protected from the atmosphere by a thin Kapton window attached to the cell edges with Vaseline. The pattern was measured on a Bruker D2 Phaser at IIT, and eventually at 11-BM at APS/ANL (Lee *et al.*, 2008; Wang *et al.*, 2008). The structure was solved and refined using the synchrotron data. Diffraction data are displayed in Fig. 4.

The synchrotron pattern was indexed with *Jade 9.5* (MDI, 2012) on a primitive triclinic unit cell having $a = 6.263$, $b = 12.029$, $c = 12.132 \text{ \AA}$, $\alpha = 74.145$, $\beta = 81.530$, $\gamma = 80.86^\circ$, and $V = 863.06 \text{ \AA}^3$. The volume corresponds to four citrate anions per cell. A Le Bail fit using this cell yielded a reduced $\chi^2 = 2.866$, but it was not possible to solve the crystal structure using this unit cell.

Removing the weak peak at 2.510° yielded a new cell, with $a = 6.131$, $b = 6.352$, $c = 12.142 \text{ \AA}$, $\alpha = 100.486$, $\beta = 98.839$, $\gamma = 110.40^\circ$, and $V = 435.852 \text{ \AA}^3$. This is a sub-cell of the original cell. The volume corresponds to two citrate anions per

cell, and space group $P\bar{1}$ was assumed. A Le Bail fit yielded a reduced χ^2 of 2.716.

The structure was solved in the sub-cell using *DASH* 3.3.2 (David *et al.*, 2006), with a citrate anion and two Na^+ cations as fragments. Two of the 25 simulated annealing runs yielded figures of merit much lower than the rest. Since two O13 atoms were 2.53 Å apart (related by a centre of symmetry), a hydrogen was placed at the centre. Refinement of this model yielded a reduced χ^2 of 2.6, but the charge did not balance. A difference-Fourier map indicated a peak 2.33 Å from O13; this is too close to be an oxygen atom, but is reasonable for an Na atom. Refinement of this model yielded a reduced χ^2 of 1.85, and an Na occupancy of 1/2.

The structure was transformed (matrix $[0\bar{1}0/2\bar{1}0/1\bar{1}1]$) to the original cell using *Materials Studio* (Dassault Systemes, 2014). The occupancies of the now two half-Na were refined. They refined to 1/0, and the low-occupancy Na moved too close to other atoms. Refinement of the resulting $\text{Na}_5\text{H}(\text{citrate})_2$ model yielded a reduced χ^2 of 1.829. This larger cell accounts for the 2.510° peak and several other very weak peaks not explained by the sub-cell. A possible C-centering, as suggested by *PLATON* (Spek, 2009), is not present.

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 C—C bonds between the terminal carboxyl carbon atoms and the adjacent carbon atoms were restrained at 1.51 (1) Å, the C—C bonds between the central carbon atoms and the adjacent carbon atoms at 1.54 (1) Å, the C—C bond between the central carbon atom and the central carboxyl carbon at 1.55 (1) Å, the C—O bond to the hydroxyl group at 1.42 (3) Å, and the C—O bonds in the carboxylate groups at 1.27 (3) Å. The tetrahedral carbon bond angles were restrained at 109 (3)°, and the angles in the planar carboxyl groups at 120 (3)°. 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 C and O atoms were constrained to be equal, and the U_{iso} values of the hydrogen atoms were constrained to be 1.3 times those of the atoms to which they are attached. A common U_{iso} value was refined for the Na atoms.

7. DFT calculations

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 eight days on a 2.4 GHz

PC. U_{iso} values were assigned to the optimized fractional coordinates based on the U_{eq} from the refined structure.

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Crystal structure of pentasodium hydrogen dicitrate from synchrotron X-ray powder diffraction data and DFT comparison

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Computing details

Program(s) used to solve structure: *DASH* (David *et al.*, 2006) for NA2HCITRATE_2_phase_1.

(NA2HCITRATE_2_phase_1) Pentasodium hydrogen dicitrate

Crystal data

$5\text{Na}^+\cdot\text{H}^+\cdot 2\text{C}_6\text{H}_5\text{O}_7^{3-}$

$M_r = 494.16$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.35262$ (9) Å

$b = 11.98628$ (18) Å

$c = 12.16544$ (16) Å

$\alpha = 73.8374$ (13)°

$\beta = 80.8808$ (15)°

$\gamma = 80.7103$ (10)°

$V = 871.72$ (2) Å³

$Z = 2$

$D_x = 1.883$ Mg m⁻³

$T = 295$ K

Data collection

11-BM APS
diffractometer

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	−0.2177 (12)	0.5862 (7)	0.8244 (8)	0.0106 (6)*
C2	0.2860 (11)	0.0939 (6)	0.8273 (7)	0.0106 (6)*
C3	−0.1531 (12)	0.6732 (7)	0.7124 (6)	0.0160 (15)*
C4	0.3818 (13)	0.1567 (7)	0.7088 (6)	0.0160 (15)*
C5	0.0541 (9)	0.7213 (5)	0.7189 (4)	0.0160 (15)*
C6	0.5481 (10)	0.2314 (5)	0.7211 (5)	0.0160 (15)*
C7	0.0934 (13)	0.8216 (7)	0.6099 (6)	0.0160 (15)*
C8	0.5470 (10)	0.3460 (6)	0.6243 (8)	0.0160 (15)*
C9	0.2718 (13)	0.8898 (7)	0.6129 (7)	0.0106 (6)*
C10	0.7654 (12)	0.3882 (8)	0.6007 (7)	0.0106 (6)*
C11	0.0204 (12)	0.7688 (6)	0.8273 (5)	0.0106 (6)*
C12	0.4917 (12)	0.2646 (7)	0.8374 (5)	0.0106 (6)*
H13	−0.28788	0.74876	0.69457	0.0209 (19)*
H14	0.24908	0.21558	0.65961	0.0209 (19)*
H15	−0.12367	0.62941	0.63910	0.0209 (19)*
H16	0.46380	0.08980	0.65996	0.0209 (19)*
H17	−0.05905	0.88634	0.59836	0.0209 (19)*

H18	0.42159	0.41571	0.65167	0.0209 (19)*
H19	0.13431	0.78343	0.53201	0.0209 (19)*
H20	0.50574	0.32991	0.54259	0.0209 (19)*
O21	−0.0775 (13)	0.5070 (8)	0.8710 (8)	0.0106 (6)*
O22	0.3882 (14)	0.0013 (7)	0.8837 (7)	0.0106 (6)*
O23	−0.4076 (12)	0.5956 (8)	0.8744 (8)	0.0106 (6)*
O24	0.0872 (12)	0.1157 (7)	0.8570 (8)	0.0106 (6)*
O25	0.3428 (14)	0.9557 (8)	0.5183 (6)	0.0106 (6)*
O26	0.8231 (13)	0.4548 (8)	0.5025 (6)	0.0106 (6)*
O27	0.3893 (13)	0.8613 (8)	0.6940 (7)	0.0106 (6)*
O28	0.8709 (14)	0.3768 (9)	0.6843 (7)	0.0106 (6)*
O29	0.1237 (15)	0.7164 (7)	0.9116 (6)	0.0106 (6)*
O30	0.6331 (15)	0.2353 (8)	0.9069 (7)	0.0106 (6)*
O31	−0.1520 (14)	0.8339 (7)	0.8448 (7)	0.0106 (6)*
O32	0.3407 (14)	0.3458 (8)	0.8468 (7)	0.0106 (6)*
O33	0.2312 (11)	0.6322 (6)	0.7256 (8)	0.0106 (6)*
O34	0.7524 (11)	0.1640 (6)	0.7170 (8)	0.0106 (6)*
H35	0.30788	0.65287	0.77888	0.0138 (8)*
H36	0.80788	0.15287	0.77888	0.0138 (8)*
Na37	0.4491 (12)	0.8035 (6)	0.8933 (6)	0.0127 (6)*
Na38	0.9726 (14)	0.3022 (6)	0.8899 (6)	0.0127 (6)*
Na39	0.2243 (13)	0.9924 (6)	0.0661 (6)	0.0127 (6)*
Na40	0.7348 (11)	0.4891 (6)	0.0844 (6)	0.0127 (6)*
Na41	0.6543 (10)	0.9874 (5)	0.6852 (5)	0.0127 (6)*
H43	0.5	1.0	0.5	0.01472*
H44	1.0	0.5	0.5	0.01472*

Geometric parameters (Å, °)

C1—C3	1.5099 (13)	O25—Na41 ^{vii}	2.375 (10)
C1—O21	1.267 (3)	O25—H43	1.168 (8)
C1—O23	1.265 (3)	O26—C10	1.274 (3)
C2—C4	1.5102 (13)	O26—H44	1.317 (8)
C2—O22	1.268 (3)	O27—C9	1.270 (3)
C2—O24	1.263 (3)	O27—Na37	2.404 (10)
C3—C1	1.5099 (13)	O27—Na41	2.409 (9)
C3—C5	1.5416 (13)	O28—C10	1.269 (3)
C3—H13	0.733 (7)	O28—Na38	2.560 (10)
C3—H15	1.216 (10)	O29—C11	1.265 (3)
C4—C2	1.5102 (13)	O29—Na37	2.416 (12)
C4—C6	1.5397 (13)	O29—Na38 ^{viii}	2.352 (10)
C4—H14	0.932 (9)	O29—Na40 ⁱⁱⁱ	2.472 (11)
C4—H16	0.966 (10)	O30—C12	1.272 (3)
C5—C3	1.5416 (13)	O30—Na37 ^{viii}	2.333 (10)
C5—C7	1.5396 (13)	O30—Na38	2.379 (12)
C5—C11	1.5483 (13)	O30—Na39 ⁱⁱⁱ	2.679 (11)
C5—O33	1.416 (3)	O31—C11	1.263 (3)
C6—C4	1.5397 (13)	O31—Na37 ⁱ	2.570 (12)

C6—C8	1.5404 (13)	O31—Na39 ^{ix}	2.551 (10)
C6—C12	1.5468 (13)	O31—Na41 ⁱ	2.590 (10)
C6—O34	1.416 (3)	O32—C12	1.268 (3)
C7—C5	1.5396 (13)	O32—Na38 ⁱ	2.422 (12)
C7—C9	1.5103 (13)	O32—Na40 ⁱⁱⁱ	2.309 (10)
C7—H17	1.206 (11)	O33—C5	1.416 (3)
C7—H19	0.817 (8)	O33—H35	0.974 (8)
C8—C6	1.5404 (13)	O33—Na40 ⁱⁱⁱ	2.386 (10)
C8—C10	1.5103 (13)	O34—C6	1.416 (3)
C8—H18	0.790 (8)	O34—H36	0.850 (9)
C8—H20	1.072 (11)	O34—Na39 ⁱⁱⁱ	2.789 (11)
C9—C7	1.5103 (13)	O34—Na41 ^{iv}	2.445 (10)
C9—O25	1.263 (3)	H35—O33	0.974 (8)
C9—O27	1.270 (3)	H36—O34	0.850 (9)
C10—C8	1.5103 (13)	Na37—O22 ^x	2.313 (11)
C10—O26	1.274 (3)	Na37—O23 ^{xi}	2.571 (11)
C10—O28	1.269 (3)	Na37—O27	2.404 (10)
C11—C5	1.5483 (13)	Na37—O29	2.416 (12)
C11—O29	1.265 (3)	Na37—O30 ^{viii}	2.333 (10)
C11—O31	1.263 (3)	Na37—O31 ^{xi}	2.570 (12)
C12—C6	1.5468 (13)	Na38—O21 ^{xi}	2.374 (12)
C12—O30	1.272 (3)	Na38—O24 ^{xi}	2.359 (11)
C12—O32	1.268 (3)	Na38—O28	2.560 (10)
H13—C3	0.733 (7)	Na38—O29 ^{viii}	2.352 (10)
H14—C4	0.932 (9)	Na38—O30	2.379 (12)
H15—C3	1.216 (10)	Na38—O32 ^{xi}	2.422 (12)
H16—C4	0.966 (10)	Na39—O22 ^{xii}	2.279 (9)
H17—C7	1.206 (11)	Na39—O22 ⁱⁱⁱ	2.646 (11)
H18—C8	0.790 (8)	Na39—O24 ^{xii}	2.758 (11)
H19—C7	0.817 (8)	Na39—O24 ^{vi}	2.457 (10)
H20—C8	1.072 (11)	Na39—O30 ⁱⁱⁱ	2.679 (11)
O21—C1	1.267 (3)	Na39—O31 ^{ix}	2.551 (10)
O21—Na38 ⁱ	2.374 (12)	Na39—O34 ⁱⁱⁱ	2.789 (11)
O21—Na40 ⁱⁱ	2.645 (10)	Na40—O21 ^{xiii}	2.645 (10)
O21—Na40 ⁱⁱⁱ	2.336 (11)	Na40—O21 ⁱⁱⁱ	2.336 (11)
O22—C2	1.268 (3)	Na40—O23 ^{xiii}	2.742 (12)
O22—Na37 ^{iv}	2.313 (11)	Na40—O23 ^{vi}	2.382 (10)
O22—Na39 ^v	2.279 (9)	Na40—O29 ⁱⁱⁱ	2.472 (11)
O22—Na39 ⁱⁱⁱ	2.646 (11)	Na40—O32 ⁱⁱⁱ	2.309 (10)
O22—Na41 ^{iv}	2.749 (10)	Na40—O33 ⁱⁱⁱ	2.386 (10)
O23—C1	1.265 (3)	Na41—O22 ^x	2.749 (10)
O23—Na37 ⁱ	2.571 (11)	Na41—O25	3.183 (11)
O23—Na40 ⁱⁱ	2.742 (12)	Na41—O25 ^{vii}	2.375 (10)
O23—Na40 ^{vi}	2.382 (10)	Na41—O27	2.409 (9)
O24—C2	1.263 (3)	Na41—O31 ^{xi}	2.590 (10)
O24—Na38 ⁱ	2.359 (11)	Na41—O34 ^x	2.445 (10)
O24—Na39 ^v	2.758 (11)	H43—O25	1.168 (8)
O24—Na39 ^{vi}	2.457 (10)	H43—O25 ^{vii}	1.168 (8)

O25—C9	1.263 (3)	H44—O26	1.317 (8)
O25—Na41	3.183 (11)	H44—O26 ^{xiv}	1.317 (8)
C3—C1—O21	119.5 (3)	C6—O34—Na41 ^{iv}	100.0 (4)
C3—C1—O23	120.5 (4)	H36—O34—Na39 ⁱⁱⁱ	38.5 (4)
O21—C1—O23	120.0 (4)	H36—O34—Na41 ^{iv}	114.5 (6)
C4—C2—O22	119.8 (3)	Na39 ⁱⁱⁱ —O34—Na41 ^{iv}	78.4 (3)
C4—C2—O24	119.1 (4)	O33—H35—Na40 ⁱⁱⁱ	98.3 (5)
O22—C2—O24	118.8 (3)	O34—H36—Na39 ⁱⁱⁱ	127.5 (5)
C1—C3—C5	110.6 (4)	O22 ^x —Na37—O23 ^{xi}	167.4 (4)
C2—C4—C6	109.1 (4)	O22 ^x —Na37—O27	85.7 (4)
C3—C5—C7	108.1 (3)	O22 ^x —Na37—O29	113.4 (4)
C3—C5—C11	108.3 (4)	O22 ^x —Na37—O30 ^{viii}	88.7 (4)
C3—C5—O33	110.6 (3)	O22 ^x —Na37—O31 ^{xi}	85.3 (3)
C7—C5—C11	109.8 (4)	O23 ^{xi} —Na37—O27	89.6 (4)
C7—C5—O33	110.0 (3)	O23 ^{xi} —Na37—O29	77.3 (4)
C11—C5—O33	110.0 (3)	O23 ^{xi} —Na37—O30 ^{viii}	99.8 (4)
C4—C6—C8	110.9 (3)	O23 ^{xi} —Na37—O31 ^{xi}	83.3 (4)
C4—C6—C12	109.7 (3)	O27—Na37—O29	81.3 (3)
C4—C6—O34	107.8 (3)	O27—Na37—O30 ^{viii}	158.3 (5)
C8—C6—C12	107.6 (4)	O27—Na37—O31 ^{xi}	92.9 (3)
C8—C6—O34	110.6 (3)	O29—Na37—O30 ^{viii}	81.8 (4)
C12—C6—O34	110.2 (5)	O29—Na37—O31 ^{xi}	159.7 (5)
C5—C7—C9	114.0 (3)	O30 ^{viii} —Na37—O31 ^{xi}	107.5 (4)
C6—C8—C10	110.0 (3)	O21 ^{xi} —Na38—O24 ^{xi}	163.1 (4)
C7—C9—O25	116.8 (5)	O21 ^{xi} —Na38—O28	80.0 (4)
C7—C9—O27	122.9 (3)	O21 ^{xi} —Na38—O29 ^{viii}	85.6 (4)
O25—C9—O27	117.4 (3)	O21 ^{xi} —Na38—O30	109.5 (5)
C8—C10—O26	119.7 (4)	O21 ^{xi} —Na38—O32 ^{xi}	78.9 (4)
C8—C10—O28	119.4 (4)	O24 ^{xi} —Na38—O28	89.3 (4)
O26—C10—O28	119.5 (4)	O24 ^{xi} —Na38—O29 ^{viii}	109.8 (4)
C5—C11—O29	119.7 (4)	O24 ^{xi} —Na38—O30	80.3 (4)
C5—C11—O31	117.9 (4)	O24 ^{xi} —Na38—O32 ^{xi}	89.8 (4)
O29—C11—O31	118.7 (4)	O28—Na38—O29 ^{viii}	148.9 (5)
C6—C12—O30	117.9 (4)	O28—Na38—O30	77.0 (4)
C6—C12—O32	118.9 (3)	O28—Na38—O32 ^{xi}	97.6 (4)
O30—C12—O32	120.0 (4)	O29 ^{viii} —Na38—O30	82.2 (4)
C1—O21—Na38 ⁱ	130.6 (10)	O29 ^{viii} —Na38—O32 ^{xi}	106.5 (4)
C1—O21—Na40 ⁱⁱ	95.2 (6)	O30—Na38—O32 ^{xi}	168.7 (5)
C1—O21—Na40 ⁱⁱⁱ	132.9 (10)	O22 ^{xii} —Na39—O22 ⁱⁱⁱ	85.5 (5)
Na38 ⁱ —O21—Na40 ⁱⁱ	94.5 (4)	O22 ^{xii} —Na39—O24 ^{xii}	50.2 (2)
Na38 ⁱ —O21—Na40 ⁱⁱⁱ	94.3 (4)	O22 ^{xii} —Na39—O24 ^{vi}	120.6 (5)
Na40 ⁱⁱ —O21—Na40 ⁱⁱⁱ	94.5 (4)	O22 ^{xii} —Na39—O30 ⁱⁱⁱ	81.4 (4)
C2—O22—Na37 ^{iv}	138.9 (9)	O22 ^{xii} —Na39—O31 ^{ix}	123.8 (4)
C2—O22—Na39 ^v	100.9 (5)	O22 ^{xii} —Na39—O34 ⁱⁱⁱ	136.3 (4)
C2—O22—Na39 ⁱⁱⁱ	119.6 (8)	O22 ⁱⁱⁱ —Na39—O24 ^{xii}	122.2 (4)
C2—O22—Na41 ^{iv}	89.3 (4)	O22 ⁱⁱⁱ —Na39—O24 ^{vi}	139.8 (4)
Na37 ^{iv} —O22—Na39 ^v	99.3 (4)	O22 ⁱⁱⁱ —Na39—O30 ⁱⁱⁱ	82.6 (4)

Na37 ^{iv} —O22—Na39 ⁱⁱⁱ	93.8 (4)	O22 ⁱⁱⁱ —Na39—O31 ^{ix}	79.2 (3)
Na37 ^{iv} —O22—Na41 ^{iv}	75.6 (3)	O22 ⁱⁱⁱ —Na39—O34 ⁱⁱⁱ	75.3 (3)
Na39 ^v —O22—Na39 ⁱⁱⁱ	94.5 (5)	O24 ^{xii} —Na39—O24 ^{vi}	97.5 (4)
Na39 ^v —O22—Na41 ^{iv}	168.6 (5)	O24 ^{xii} —Na39—O30 ⁱⁱⁱ	117.4 (3)
Na39 ⁱⁱⁱ —O22—Na41 ^{iv}	75.9 (3)	O24 ^{xii} —Na39—O31 ^{ix}	95.5 (3)
C1—O23—Na37 ⁱ	113.8 (6)	O24 ^{xii} —Na39—O34 ⁱⁱⁱ	162.2 (4)
C1—O23—Na40 ⁱⁱ	90.8 (7)	O24 ^{vi} —Na39—O30 ⁱⁱⁱ	72.9 (4)
C1—O23—Na40 ^{vi}	143.0 (9)	O24 ^{vi} —Na39—O31 ^{ix}	104.8 (4)
Na37 ⁱ —O23—Na40 ⁱⁱ	102.4 (4)	O24 ^{vi} —Na39—O34 ⁱⁱⁱ	64.7 (3)
Na37 ⁱ —O23—Na40 ^{vi}	100.1 (4)	O30 ⁱⁱⁱ —Na39—O31 ^{ix}	147.1 (4)
Na40 ⁱⁱ —O23—Na40 ^{vi}	96.1 (3)	O30 ⁱⁱⁱ —Na39—O34 ⁱⁱⁱ	57.7 (3)
C2—O24—Na38 ⁱ	115.7 (6)	O31 ^{ix} —Na39—O34 ⁱⁱⁱ	91.0 (3)
C2—O24—Na39 ^v	79.2 (5)	O21 ^{xiii} —Na40—O21 ⁱⁱⁱ	85.5 (4)
C2—O24—Na39 ^{vi}	138.3 (6)	O21 ^{xiii} —Na40—O23 ^{xiii}	47.98 (16)
Na38 ⁱ —O24—Na39 ^v	101.1 (4)	O21 ^{xiii} —Na40—O23 ^{vi}	113.9 (5)
Na38 ⁱ —O24—Na39 ^{vi}	104.4 (4)	O21 ^{xiii} —Na40—O29 ⁱⁱⁱ	77.7 (3)
Na39 ^v —O24—Na39 ^{vi}	82.5 (4)	O21 ^{xiii} —Na40—O32 ⁱⁱⁱ	119.2 (4)
C9—O27—Na37	153.7 (7)	O21 ^{xiii} —Na40—O33 ⁱⁱⁱ	139.4 (4)
C9—O27—Na41	117.3 (4)	O21 ⁱⁱⁱ —Na40—O23 ^{xiii}	124.8 (4)
Na37—O27—Na41	80.9 (3)	O21 ⁱⁱⁱ —Na40—O23 ^{vi}	150.1 (4)
C10—O28—Na38	157.8 (5)	O21 ⁱⁱⁱ —Na40—O29 ⁱⁱⁱ	82.7 (4)
C11—O29—Na37	109.6 (7)	O21 ⁱⁱⁱ —Na40—O32 ⁱⁱⁱ	82.0 (4)
C11—O29—Na38 ^{viii}	130.2 (6)	O21 ⁱⁱⁱ —Na40—O33 ⁱⁱⁱ	71.4 (4)
C11—O29—Na40 ⁱⁱⁱ	114.1 (5)	O23 ^{xiii} —Na40—O23 ^{vi}	83.9 (3)
Na37—O29—Na38 ^{viii}	96.9 (4)	O23 ^{xiii} —Na40—O29 ⁱⁱⁱ	108.4 (3)
Na37—O29—Na40 ⁱⁱⁱ	102.0 (4)	O23 ^{xiii} —Na40—O32 ⁱⁱⁱ	95.5 (4)
Na38 ^{viii} —O29—Na40 ⁱⁱⁱ	99.8 (4)	O23 ^{xiii} —Na40—O33 ⁱⁱⁱ	163.3 (4)
C12—O30—Na37 ^{viii}	123.5 (6)	O23 ^{vi} —Na40—O29 ⁱⁱⁱ	79.9 (4)
C12—O30—Na38	129.9 (7)	O23 ^{vi} —Na40—O32 ⁱⁱⁱ	105.3 (5)
C12—O30—Na39 ⁱⁱⁱ	108.7 (6)	O23 ^{vi} —Na40—O33 ⁱⁱⁱ	79.5 (4)
Na37 ^{viii} —O30—Na38	98.5 (4)	O29 ⁱⁱⁱ —Na40—O32 ⁱⁱⁱ	156.1 (4)
Na37 ^{viii} —O30—Na39 ⁱⁱⁱ	88.3 (4)	O29 ⁱⁱⁱ —Na40—O33 ⁱⁱⁱ	66.9 (3)
Na38—O30—Na39 ⁱⁱⁱ	97.4 (4)	O32 ⁱⁱⁱ —Na40—O33 ⁱⁱⁱ	90.8 (4)
C11—O31—Na37 ⁱ	134.6 (8)	O22 ^x —Na41—O25 ^{vii}	140.9 (4)
C11—O31—Na39 ^{ix}	130.3 (7)	O22 ^x —Na41—O27	76.6 (4)
C11—O31—Na41 ⁱ	124.9 (5)	O22 ^x —Na41—O31 ^{xi}	76.7 (3)
Na37 ⁱ —O31—Na39 ^{ix}	90.2 (3)	O22 ^x —Na41—O34 ^x	79.3 (3)
Na37 ⁱ —O31—Na41 ⁱ	74.5 (3)	O25 ^{vii} —Na41—O27	88.0 (4)
Na39 ^{ix} —O31—Na41 ⁱ	80.4 (3)	O25 ^{vii} —Na41—O31 ^{xi}	140.6 (4)
C12—O32—Na38 ⁱ	119.5 (8)	O25 ^{vii} —Na41—O34 ^x	100.5 (4)
C12—O32—Na40 ⁱⁱⁱ	140.3 (7)	O27—Na41—O31 ^{xi}	92.3 (4)
Na38 ⁱ —O32—Na40 ⁱⁱⁱ	93.7 (4)	O27—Na41—O34 ^x	150.5 (4)
C5—O33—Na40 ⁱⁱⁱ	114.0 (4)	O31 ^{xi} —Na41—O34 ^x	98.4 (4)
H35—O33—Na40 ⁱⁱⁱ	57.8 (5)	O25—H43—O25 ^{vii}	180.0
C6—O34—Na39 ⁱⁱⁱ	106.7 (5)	O26—H44—O26 ^{xiv}	180.0

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

(NA2HCITRATE_2_phase_2) Silicon

Crystal data

Si	$V = 160.20 (1) \text{ \AA}^3$
$M_r = 28.09$	$Z = 8$
Cubic, $Fd\bar{3}m$	$D_x = 2.329 \text{ Mg m}^{-3}$
Hall symbol: -F 4vw 2vw	$T = 295 \text{ K}$
$a = 5.431046 (9) \text{ \AA}$	

Data collection

$$2\theta_{\min} = 0.5^\circ, 2\theta_{\max} = 50.0^\circ, 2\theta_{\text{step}} = 0.001^\circ$$

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Si1	0.125	0.125	0.125	0.0056 (3)*

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

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 (1)	Si1 ⁱⁱ —Si1—Si1 ⁱⁱⁱ	109.4712 (1)
Si1 ⁱ —Si1—Si1 ⁱⁱⁱ	109.4712 (1)	Si1 ⁱⁱ —Si1—Si1 ^{iv}	109.4712 (1)
Si1 ⁱ —Si1—Si1 ^{iv}	109.4712 (1)	Si1 ⁱⁱⁱ —Si1—Si1 ^{iv}	109.4712 (1)

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) $-x, -y, -z$.

(Na5Hcit2_DFT)

Crystal data

$\text{Na}_5\text{H}(\text{C}_6\text{H}_5\text{O}_7)_2$	$\alpha = 73.8374^\circ$
$M_r = 494.13$	$\beta = 80.8808^\circ$
Triclinic, $P\bar{1}$	$\gamma = 80.7103^\circ$
Hall symbol: -P 1	$V = 871.72 \text{ \AA}^3$
$a = 6.3526 \text{ \AA}$	$Z = 2$
$b = 11.9863 \text{ \AA}$	$D_x = 1.883 \text{ Mg m}^{-3}$
$c = 12.1654 \text{ \AA}$	$T = 295 \text{ K}$

Data collection

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.21093	0.58380	0.83744	0.01060*
C2	0.28547	0.08699	0.82748	0.01060*
C3	-0.11904	0.65713	0.72068	0.01600*
C4	0.34300	0.18316	0.71790	0.01600*
C5	0.06950	0.72103	0.72811	0.01600*
C6	0.53786	0.24180	0.72599	0.01600*

C7	0.11665	0.81156	0.61196	0.01600*
C8	0.55181	0.35732	0.63154	0.01600*
C9	0.29966	0.88149	0.60472	0.01060*
C10	0.77028	0.40070	0.61710	0.01060*
C11	0.00917	0.77940	0.83069	0.01060*
C12	0.50875	0.26301	0.84778	0.01060*
O21	−0.08985	0.50169	0.89446	0.01060*
O22	0.43125	0.00445	0.86367	0.01060*
O23	−0.40674	0.61148	0.87458	0.01060*
O24	0.09817	0.09733	0.88046	0.01060*
O25	0.34064	0.95546	0.50492	0.01060*
O26	0.82824	0.46330	0.51541	0.01060*
O27	0.40445	0.86921	0.68646	0.01060*
O28	0.87727	0.37570	0.70033	0.01060*
O29	0.11832	0.74256	0.91351	0.01060*
O30	0.65064	0.21054	0.91295	0.01060*
O31	−0.15265	0.85830	0.82412	0.01060*
O32	0.34170	0.32615	0.87533	0.01060*
O33	0.25360	0.63635	0.74506	0.01060*
O34	0.72908	0.16202	0.71125	0.01060*
H35	0.36530	0.67271	0.76366	0.01380*
H36	0.83443	0.18009	0.75138	0.01380*
Na37	0.46548	0.79769	0.89886	0.01270*
Na38	0.97393	0.30775	0.88552	0.01270*
Na39	0.22004	0.98848	0.06887	0.01270*
Na40	0.71654	0.49717	0.07535	0.01270*
Na41	0.66889	0.98575	0.69266	0.01270*
H13	−0.24775	0.72123	0.68279	0.02090*
H15	−0.05830	0.59958	0.66395	0.02090*
H14	0.62099	0.85243	0.35789	0.02090*
H16	0.79387	0.74936	0.29832	0.02090*
H18	0.47857	0.65251	0.45109	0.02090*
H20	0.57361	0.57501	0.34726	0.02090*
H17	−0.16193	0.23620	0.45478	0.02090*
H19	0.02421	0.12681	0.41491	0.02090*
H43	0.50000	1.00000	0.50000	0.01470*
H44	1.00000	0.50000	0.50000	0.01470*

Bond lengths (Å)

C1—C3	1.529	C8—H18 ⁱ	1.094
C1—O21	1.259	C8—H20 ⁱ	1.096
C1—O23	1.278	C9—O25	1.305
C2—C4	1.538	C9—O27	1.245
C2—O22	1.273	C10—O26	1.287
C2—O24	1.265	C10—O28	1.249
C3—C5	1.549	C11—O29	1.251
C3—H13	1.091	C11—O31	1.276

C3—H15	1.094	C12—O30	1.266
C4—C6	1.547	C12—O32	1.257
C4—H14 ⁱ	1.098	O25—H43	1.205
C4—H16 ⁱ	1.090	O26—H44	1.210
C5—C7	1.543	O33—H35	0.975
C5—C11	1.563	O34—H36	0.971
C5—O33	1.420	H14—C4 ⁱ	1.098
C6—C8	1.539	H16—C4 ⁱ	1.090
C6—C12	1.550	H18—C8 ⁱ	1.094
C6—O34	1.440	H20—C8 ⁱ	1.096
C7—C9	1.518	H17—C7 ⁱⁱ	1.096
C7—H17 ⁱⁱ	1.096	H19—C7 ⁱⁱ	1.095
C7—H19 ⁱⁱ	1.095	H43—O25 ⁱⁱⁱ	1.205
C8—C10	1.527	H44—O26 ^{iv}	1.210

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O26—H44 \cdots O26 ^{iv}	1.210	1.210	2.419	180.0
O25—H43 \cdots O25 ⁱⁱⁱ	1.205	1.205	2.409	180.0
O33—H35 \cdots O23 ^v	0.975	2.045	2.799	132.8
O33—H35 \cdots O29	0.975	2.450	2.664	91.7
O34—H36 \cdots O30	0.971	2.212	2.631	104.6
O34—H36 \cdots O28	0.971	2.302	2.831	113.4
O34—H36 \cdots O24 ^v	0.971	2.385	3.223	144.3

Symmetry codes: (iii) $-x+1, -y+2, -z+1$; (iv) $-x+2, -y+1, -z+1$; (v) $x+1, y, z$.