

Poly[(μ_5 -5-carboxylatotetrahydrofuran-2,3,4-tricarboxylic acid)sodium]

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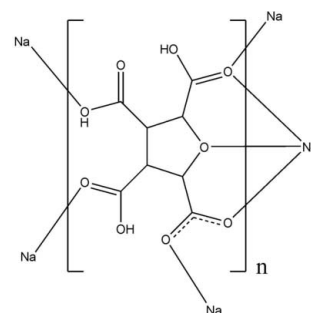
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.032; wR factor = 0.090; data-to-parameter ratio = 13.8.

The search for the novel metal-organic frameworks (MOFs) materials using tetrahydrofuran-2,3,4,5-tetracarboxylic acid (THFTCA) as a versatile multi-carboxyl ligand, lead to the synthesis and the structure determination of the title compound, $[\text{Na}(\text{H}_3\text{THFTCA})]$ or $[\text{Na}(\text{C}_8\text{H}_7\text{O}_9)]_n$, which was obtained by a solution reaction at room temperature. The ligand is mono-deprotonated, coordinating five sodium ions through one furan oxygen atom and six carboxyl oxygen atoms. The sodium ion exhibits a distorted pentagonal-bipyramidal NaO_7 geometry consisting of seven O atoms derived from five surrounding ligands. Two adjacent pentagonal bipyramids share an O—O edge, forming a dinuclear sodium cluster. Finally, these clusters are effectively linked by the carboxyl groups of THFTCA ligands, forming a firm metal organic framework and O—H \cdots O hydrogen bonds contribute to the crystal packing.

Related literature

For potential applications of metal-organic frameworks (MOFs), see: Moulton & Zaworotko (2001); Bradshaw *et al.* (2007). Self-assembly of selected ligands around d -transition metal ions is a widespread method for obtaining novel MOF structures, see: Leininger *et al.* (2000). In contrast, the s -elements are more flexible of their coordination behaviour, and maybe present in more various structures, see: Lu *et al.* (2007). For related MOF materials constructed from the THFTCA ligand, see: Hanson *et al.* (2004); Thuéry *et al.* (2004); Ai *et al.* (2008); Wang & Sevov (2007); Wang *et al.* (2007); Lü (2008). For related s -elements and THFTCA ligand compound structures, see: Barnes & Paton (1984) for Cs^+ and Ca^{2+} ; Barnes (2002) for Na^+ ; Paul & Martin (1967) for Rb^+ .



Experimental

Crystal data

$[\text{Na}(\text{C}_8\text{H}_7\text{O}_9)]$

$M_r = 270.13$

Monoclinic, $P2_1/c$

$a = 8.0663$ (16) Å

$b = 13.417$ (3) Å

$c = 9.7358$ (19) Å

$\beta = 109.90$ (3)°

$V = 990.7$ (3) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.20$ mm⁻¹

$T = 296$ K

$0.41 \times 0.28 \times 0.10$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.921$, $T_{\max} = 0.980$

9567 measured reflections

2263 independent reflections

2095 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.090$

$S = 1.09$

2263 reflections

164 parameters

H-atom parameters constrained

$\Delta\rho_{\max} = 0.39$ e Å⁻³

$\Delta\rho_{\min} = -0.32$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O3}-\text{H3}\cdots\text{O9}^{\text{i}}$	0.84	1.70	2.5395 (15)	173
$\text{O5}-\text{H5}\cdots\text{O6}^{\text{ii}}$	0.84	1.83	2.6468 (16)	165
$\text{O7}-\text{H7}\cdots\text{O8}^{\text{iii}}$	0.84	1.68	2.5169 (14)	171

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We are grateful for financial support from the National Natural Science Foundation of China (projects 50702054 and 20803070) and the Analysis and Testing Foundation of Zhejiang Province (projects 2008 F70034 and 2008 F70053).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2234).

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supplementary materials

Acta Cryst. (2009). E65, m1419-m1420 [doi:10.1107/S160053680904269X]

Poly[(μ_5 -5-carboxylatotetrahydrofuran-2,3,4-tricarboxylic acid)sodium]

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Comment

Metal organic frameworks (MOFs) have attracted a great deal of interest owing to the ability to tune their porosity and the functionalities that are incorporated within the framework scaffolds. As a result, numerous MOFs have been engineered for a number of potential applications, including gas storage, nonlinear optics, catalysis, and so on. (Moulton *et al.* 2001; Bradshaw *et al.* 2007) Usually, highly directional coordination bonds are adopted in the design of MOFs, and self-assembly of selected ligands around d-transition metal ions is now a widespread method for obtaining novel MOFs structure (Leininger *et al.* 2000). In contrast, the s-elements are more flexible of their coordination behaviour, and maybe present in more various structures (Lu *et al.* 2007). On the other hand, for the complex ligand with large numbers of potential binding sites, such as, tetrahydrofuran-2, 3, 4, 5-tetracarboxylic acid, it is difficult to predict the final structure. Therefore, the investigation of these complex ligands might provide novel MOFs with interesting structural topology. However, reports on THFTCA are rare (Hanson *et al.* 2004). Here, we report a three-dimensional MOFs compound Na(H₃THFTCA) (I), which is assembled from THFTCA and sodium ion.

The title compound has a three-dimensional framework structure constructed by mono deprotonated THFTCA ligand; the asymmetric unit contains one full chiral THFTCA ligand and one sodium atom (Fig. 1). The THFTCA ligand coordinates the sodium ion with its furan oxygen atom and two adjacent carboxyl oxygen atoms, while its four carboxyl groups also grasp the neighbouring four sodium ions (Scheme 1). Thus, the sodium ion is located in a distorted pentagonal bipyramid NaO₇, coordinated by seven O atoms from the five ligands. The two pentagonal bipyramids are fused via a common edge O2—O2, generating a dinuclear sodium cluster with an inversion centre at the midpoint of edge O2—O2 (Fig. 2). The title compound crystallizes in the centrosymmetric space group P2₁/c implying the presence of a racemate (1:1) in the crystal. The dinuclear sodium clusters are connected by carboxyl groups of THFTCA ligand. The crystal packing includes firm framework of multi-carboxyl ligand and sodium ion connected by hydrogen bonds of O—H \cdots O (Table 1, Fig. 3).

Experimental

All chemicals were obtained from commercial sources and were used as obtained. The title compound was handily synthesized by a solution reaction from H₄THFTCA. H₄THFTCA (154 mg, 0.6 mmol) and NaOH (25 mg, 0.6 mmol) was dissolved in 10 ml of water. To this solution was added a 5 ml aqueous solution of Nd(NO₃)₃·6H₂O (89 mg, 0.2 mmol) at room temperature. Amount of colourless crystals were obtained after the filtration was slowly evaporated at room temperature for several days.

Refinement

The structure was solved using direct methods and refined by full-matrix least-squares techniques. All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. All hydrogen atoms were added at calculated positions and refined using a riding model. (Sheldrick *et al.*, 2008).

Figures

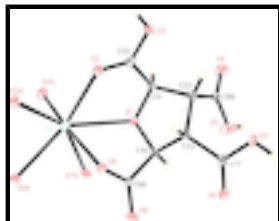


Fig. 1. Structure and labeling of the title compound, with displacement ellipsoids drawn at the 30% probability level and H atoms shown as small spheres of arbitrary radii.

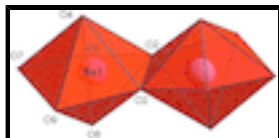


Fig. 2. The distorted pentagonal bipyramid NaO_7 coordination polyhedron with the common edge O2-O2 shows a dinuclear sodium unit.

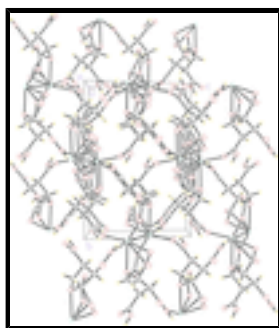


Fig. 3. The packing diagram viewed along the axis a , Na: blue diagonal; O: red inner dot; C: black circles; and H: small green circles.

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Crystal data

$[\text{Na}(\text{C}_8\text{H}_7\text{O}_9)]$

$M_r = 270.13$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 8.0663\ (16)\ \text{\AA}$

$b = 13.417\ (3)\ \text{\AA}$

$c = 9.7358\ (19)\ \text{\AA}$

$\beta = 109.90\ (3)^\circ$

$V = 990.7\ (3)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 552$

$D_x = 1.811\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71075\ \text{\AA}$

Cell parameters from 8534 reflections

$\theta = 3.0\text{--}27.4^\circ$

$\mu = 0.20\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Platelet, colorless

$0.41 \times 0.28 \times 0.10\ \text{mm}$

Data collection

Rigaku R-Axis RAPID
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: $14.6306\ \text{pixels mm}^{-1}$

$T = 296\ \text{K}$

CCD_Profile_fitting scans

2263 independent reflections

2095 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\text{max}} = 27.4^\circ$

$\theta_{\text{min}} = 3.0^\circ$

$h = -10 \rightarrow 10$

Absorption correction: Multi-scan
(ABSCOR; Higashi, 1995) $k = -17 \rightarrow 17$
 $T_{\min} = 0.921$, $T_{\max} = 0.980$ $l = -12 \rightarrow 12$
9567 measured reflections

Refinement

Refinement on F^2 Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.4297P]$
 $R[F^2 > 2\sigma(F^2)] = 0.032$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.090$ $(\Delta/\sigma)_{\max} < 0.001$
 $S = 1.09$ $\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$
2263 reflections $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$
164 parameters Extinction correction: SHELXL,
 $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.034 (3)
Secondary atom site location: difference Fourier map

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Na1	0.16477 (7)	0.01271 (4)	0.39826 (6)	0.02678 (17)
O1	0.19961 (11)	0.20181 (7)	0.39121 (9)	0.0179 (2)
O2	-0.03876 (14)	0.10910 (7)	0.48915 (13)	0.0313 (3)
O3	-0.17693 (14)	0.25311 (8)	0.49566 (13)	0.0333 (3)
H3	-0.2454	0.2189	0.5252	0.033*
O4	0.02774 (14)	0.47842 (9)	0.33101 (13)	0.0362 (3)
O5	0.31727 (15)	0.46326 (10)	0.42755 (16)	0.0506 (4)
H5	0.3204	0.5163	0.3827	0.051*
O6	0.60941 (12)	0.37079 (7)	0.68721 (11)	0.0251 (2)
O7	0.39834 (13)	0.43913 (7)	0.75861 (11)	0.0281 (2)
H7	0.4778	0.4798	0.8028	0.028*
O8	0.39053 (12)	0.07324 (7)	0.60685 (11)	0.0234 (2)

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O9	0.63546 (12)	0.13823 (7)	0.58976 (12)	0.0272 (2)
C11	0.05765 (15)	0.26035 (9)	0.40594 (13)	0.0167 (2)
H11	-0.0167	0.2819	0.3091	0.017*
C22	0.14282 (15)	0.34871 (9)	0.50496 (13)	0.0159 (2)
H22	0.0684	0.3706	0.5593	0.016*
C33	0.31740 (15)	0.30122 (8)	0.60199 (12)	0.0147 (2)
H33	0.2877	0.2598	0.6734	0.015*
C44	0.36719 (15)	0.23349 (8)	0.49541 (12)	0.0149 (2)
H44	0.4337	0.2705	0.4470	0.015*
C55	-0.05776 (16)	0.19811 (10)	0.46899 (14)	0.0202 (3)
C66	0.15670 (16)	0.43746 (9)	0.41175 (14)	0.0197 (3)
C77	0.45783 (16)	0.37456 (9)	0.68641 (13)	0.0178 (2)
C88	0.47422 (15)	0.14121 (9)	0.56952 (13)	0.0163 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0233 (3)	0.0215 (3)	0.0317 (3)	-0.0036 (2)	0.0044 (2)	-0.0026 (2)
O1	0.0145 (4)	0.0192 (4)	0.0191 (4)	0.0008 (3)	0.0043 (3)	-0.0038 (3)
O2	0.0268 (5)	0.0179 (5)	0.0538 (7)	0.0001 (4)	0.0195 (5)	0.0058 (4)
O3	0.0272 (5)	0.0212 (5)	0.0619 (7)	-0.0005 (4)	0.0286 (5)	0.0012 (5)
O4	0.0261 (5)	0.0321 (6)	0.0424 (6)	0.0028 (4)	0.0012 (5)	0.0170 (5)
O5	0.0222 (6)	0.0509 (7)	0.0725 (9)	-0.0036 (5)	0.0078 (6)	0.0436 (7)
O6	0.0173 (5)	0.0233 (5)	0.0327 (5)	-0.0026 (4)	0.0059 (4)	0.0003 (4)
O7	0.0258 (5)	0.0243 (5)	0.0355 (5)	-0.0092 (4)	0.0123 (4)	-0.0145 (4)
O8	0.0186 (4)	0.0189 (4)	0.0319 (5)	0.0009 (4)	0.0077 (4)	0.0092 (4)
O9	0.0158 (4)	0.0232 (5)	0.0433 (6)	0.0031 (4)	0.0112 (4)	0.0073 (4)
C11	0.0146 (5)	0.0151 (5)	0.0195 (5)	0.0006 (4)	0.0047 (4)	0.0002 (4)
C22	0.0140 (5)	0.0138 (5)	0.0199 (5)	-0.0006 (4)	0.0056 (4)	0.0000 (4)
C33	0.0154 (5)	0.0128 (5)	0.0161 (5)	-0.0004 (4)	0.0058 (4)	0.0007 (4)
C44	0.0142 (5)	0.0142 (5)	0.0164 (5)	-0.0006 (4)	0.0052 (4)	0.0012 (4)
C55	0.0152 (5)	0.0188 (6)	0.0256 (6)	-0.0023 (5)	0.0057 (5)	-0.0010 (5)
C66	0.0190 (6)	0.0151 (5)	0.0238 (6)	0.0001 (4)	0.0057 (5)	0.0014 (4)
C77	0.0197 (6)	0.0149 (5)	0.0171 (5)	-0.0017 (4)	0.0042 (4)	0.0021 (4)
C88	0.0158 (5)	0.0149 (5)	0.0182 (5)	0.0009 (4)	0.0058 (4)	-0.0008 (4)

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

Na1—O4 ⁱ	2.2903 (14)	O7—C77	1.3056 (16)
Na1—O8	2.3626 (14)	O7—Na1 ^{vi}	2.7495 (13)
Na1—O2 ⁱⁱ	2.3800 (12)	O7—H7	0.8400
Na1—O2	2.4778 (13)	O8—C88	1.2595 (15)
Na1—O1	2.5561 (12)	O9—C88	1.2480 (15)
Na1—O9 ⁱⁱⁱ	2.5658 (12)	O9—Na1 ⁱⁱⁱ	2.5658 (12)
Na1—O7 ^{iv}	2.7495 (13)	C11—C55	1.5264 (17)
O1—C11	1.4354 (14)	C11—C22	1.5349 (16)
O1—C44	1.4502 (14)	C11—H11	0.9734
O2—C55	1.2114 (16)	C22—C66	1.5242 (17)

O2—Na1 ⁱⁱ	2.3800 (12)	C22—C33	1.5413 (16)
O3—C55	1.3061 (16)	C22—H22	0.9710
O3—H3	0.8400	C33—C77	1.5143 (16)
O4—C66	1.2021 (17)	C33—C44	1.5322 (16)
O4—Na1 ^v	2.2903 (14)	C33—H33	0.9811
O5—C66	1.2982 (17)	C44—C88	1.5410 (16)
O5—H5	0.8402	C44—H44	0.9638
O6—C77	1.2210 (16)		
O4 ⁱ —Na1—O8	167.22 (5)	O1—C11—C22	106.48 (9)
O4 ⁱ —Na1—O2 ⁱⁱ	93.25 (5)	C55—C11—C22	111.95 (10)
O8—Na1—O2 ⁱⁱ	99.51 (4)	O1—C11—H11	108.4
O4 ⁱ —Na1—O2	98.11 (5)	C55—C11—H11	107.0
O8—Na1—O2	85.65 (4)	C22—C11—H11	112.0
O2 ⁱⁱ —Na1—O2	75.83 (4)	C66—C22—C11	109.71 (10)
O4 ⁱ —Na1—O1	102.56 (4)	C66—C22—C33	116.80 (10)
O8—Na1—O1	67.81 (3)	C11—C22—C33	100.71 (9)
O2 ⁱⁱ —Na1—O1	139.65 (4)	C66—C22—H22	105.8
O2—Na1—O1	65.41 (3)	C11—C22—H22	110.4
O4 ⁱ —Na1—O9 ⁱⁱⁱ	95.32 (5)	C33—C22—H22	113.4
O8—Na1—O9 ⁱⁱⁱ	86.78 (4)	C77—C33—C44	115.65 (10)
O2 ⁱⁱ —Na1—O9 ⁱⁱⁱ	78.30 (4)	C77—C33—C22	114.99 (10)
O2—Na1—O9 ⁱⁱⁱ	151.39 (4)	C44—C33—C22	103.04 (9)
O1—Na1—O9 ⁱⁱⁱ	135.36 (4)	C77—C33—H33	107.5
O4 ⁱ —Na1—O7 ^{iv}	85.22 (5)	C44—C33—H33	109.1
O8—Na1—O7 ^{iv}	83.50 (4)	C22—C33—H33	106.0
O2 ⁱⁱ —Na1—O7 ^{iv}	149.44 (4)	O1—C44—C33	104.52 (9)
O2—Na1—O7 ^{iv}	134.65 (4)	O1—C44—C88	109.41 (9)
O1—Na1—O7 ^{iv}	69.72 (3)	C33—C44—C88	113.18 (9)
O9 ⁱⁱⁱ —Na1—O7 ^{iv}	71.49 (4)	O1—C44—H44	110.5
C11—O1—C44	110.80 (9)	C33—C44—H44	110.1
C11—O1—Na1	116.21 (7)	C88—C44—H44	109.1
C44—O1—Na1	110.91 (6)	O2—C55—O3	125.86 (12)
C55—O2—Na1 ⁱⁱ	134.52 (9)	O2—C55—C11	122.93 (12)
C55—O2—Na1	121.28 (9)	O3—C55—C11	111.21 (11)
Na1 ⁱⁱ —O2—Na1	104.17 (4)	O4—C66—O5	124.19 (13)
C55—O3—H3	111.9	O4—C66—C22	121.59 (12)
C66—O4—Na1 ^v	151.37 (11)	O5—C66—C22	114.22 (11)
C66—O5—H5	111.7	O6—C77—O7	125.09 (12)
C77—O7—Na1 ^{vi}	149.49 (8)	O6—C77—C33	122.61 (11)
C77—O7—H7	110.5	O7—C77—C33	112.29 (11)
Na1 ^{vi} —O7—H7	97.2	O9—C88—O8	124.29 (11)
C88—O8—Na1	109.44 (8)	O9—C88—C44	119.16 (11)
C88—O9—Na1 ⁱⁱⁱ	129.58 (8)	O8—C88—C44	116.55 (10)

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O1—C11—C55	110.94 (10)		
O4 ⁱ —Na1—O1—C11	77.60 (8)	C11—C22—C33—C77	164.09 (10)
O8—Na1—O1—C11	-111.21 (8)	C66—C22—C33—C44	-81.34 (12)
O2 ⁱⁱ —Na1—O1—C11	-33.01 (10)	C11—C22—C33—C44	37.35 (11)
O2—Na1—O1—C11	-15.66 (7)	C11—O1—C44—C33	12.61 (12)
O9 ⁱⁱⁱ —Na1—O1—C11	-171.25 (7)	Na1—O1—C44—C33	-117.99 (7)
O7 ^{iv} —Na1—O1—C11	157.62 (8)	C11—O1—C44—C88	134.10 (10)
O4 ⁱ —Na1—O1—C44	-154.68 (7)	Na1—O1—C44—C88	3.50 (10)
O8—Na1—O1—C44	16.50 (7)	C77—C33—C44—O1	-157.88 (9)
O2 ⁱⁱ —Na1—O1—C44	94.70 (9)	C22—C33—C44—O1	-31.56 (11)
O2—Na1—O1—C44	112.05 (8)	C77—C33—C44—C88	83.16 (12)
O9 ⁱⁱⁱ —Na1—O1—C44	-43.54 (9)	C22—C33—C44—C88	-150.52 (10)
O7 ^{iv} —Na1—O1—C44	-74.67 (7)	Na1 ⁱⁱ —O2—C55—O3	-7.4 (2)
O4 ⁱ —Na1—O2—C55	-87.32 (12)	Na1—O2—C55—O3	170.75 (11)
O8—Na1—O2—C55	80.39 (11)	Na1 ⁱⁱ —O2—C55—C11	173.27 (9)
O2 ⁱⁱ —Na1—O2—C55	-178.65 (14)	Na1—O2—C55—C11	-8.56 (18)
O1—Na1—O2—C55	12.84 (10)	O1—C11—C55—O2	-6.66 (17)
O9 ⁱⁱⁱ —Na1—O2—C55	155.50 (10)	C22—C11—C55—O2	-125.45 (14)
O7 ^{iv} —Na1—O2—C55	3.96 (14)	O1—C11—C55—O3	173.94 (10)
O4 ⁱ —Na1—O2—Na1 ⁱⁱ	91.33 (5)	C22—C11—C55—O3	55.15 (14)
O8—Na1—O2—Na1 ⁱⁱ	-100.96 (5)	Na1 ^v —O4—C66—O5	73.4 (3)
O2 ⁱⁱ —Na1—O2—Na1 ⁱⁱ	0.0	Na1 ^v —O4—C66—C22	-106.3 (2)
O1—Na1—O2—Na1 ⁱⁱ	-168.51 (6)	C11—C22—C66—O4	65.15 (16)
O9 ⁱⁱⁱ —Na1—O2—Na1 ⁱⁱ	-25.85 (10)	C33—C22—C66—O4	178.86 (12)
O7 ^{iv} —Na1—O2—Na1 ⁱⁱ	-177.39 (5)	C11—C22—C66—O5	-114.57 (14)
O4 ⁱ —Na1—O8—C88	3.2 (2)	C33—C22—C66—O5	-0.86 (17)
O2 ⁱⁱ —Na1—O8—C88	-179.35 (8)	Na1 ^{vi} —O7—C77—O6	-151.33 (12)
O2—Na1—O8—C88	-104.52 (9)	Na1 ^{vi} —O7—C77—C33	27.3 (2)
O1—Na1—O8—C88	-39.34 (8)	C44—C33—C77—O6	-10.65 (16)
O9 ⁱⁱⁱ —Na1—O8—C88	103.09 (9)	C22—C33—C77—O6	-130.65 (12)
O7 ^{iv} —Na1—O8—C88	31.37 (8)	C44—C33—C77—O7	170.70 (10)
C44—O1—C11—C55	-110.30 (11)	C22—C33—C77—O7	50.70 (14)
Na1—O1—C11—C55	17.46 (12)	Na1 ⁱⁱⁱ —O9—C88—O8	29.64 (19)
C44—O1—C11—C22	11.74 (12)	Na1 ⁱⁱⁱ —O9—C88—C44	-150.88 (8)
Na1—O1—C11—C22	139.51 (7)	Na1—O8—C88—O9	-120.38 (12)
O1—C11—C22—C66	93.14 (11)	Na1—O8—C88—C44	60.13 (12)
C55—C11—C22—C66	-145.46 (10)	O1—C44—C88—O9	138.01 (11)
O1—C11—C22—C33	-30.58 (11)	C33—C44—C88—O9	-105.88 (13)
C55—C11—C22—C33	90.82 (11)	O1—C44—C88—O8	-42.47 (14)
C66—C22—C33—C77	45.40 (14)	C33—C44—C88—O8	73.63 (13)

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

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 \cdots O9 ^{vii}	0.84	1.70	2.5395 (15)	173
O5—H5 \cdots O6 ^{viii}	0.84	1.83	2.6468 (16)	165
O7—H7 \cdots O8 ^{ix}	0.84	1.68	2.5169 (14)	171

Symmetry codes: (vii) $x-1, y, z$; (viii) $-x+1, -y+1, -z+1$; (ix) $-x+1, y+1/2, -z+3/2$.

Fig. 1

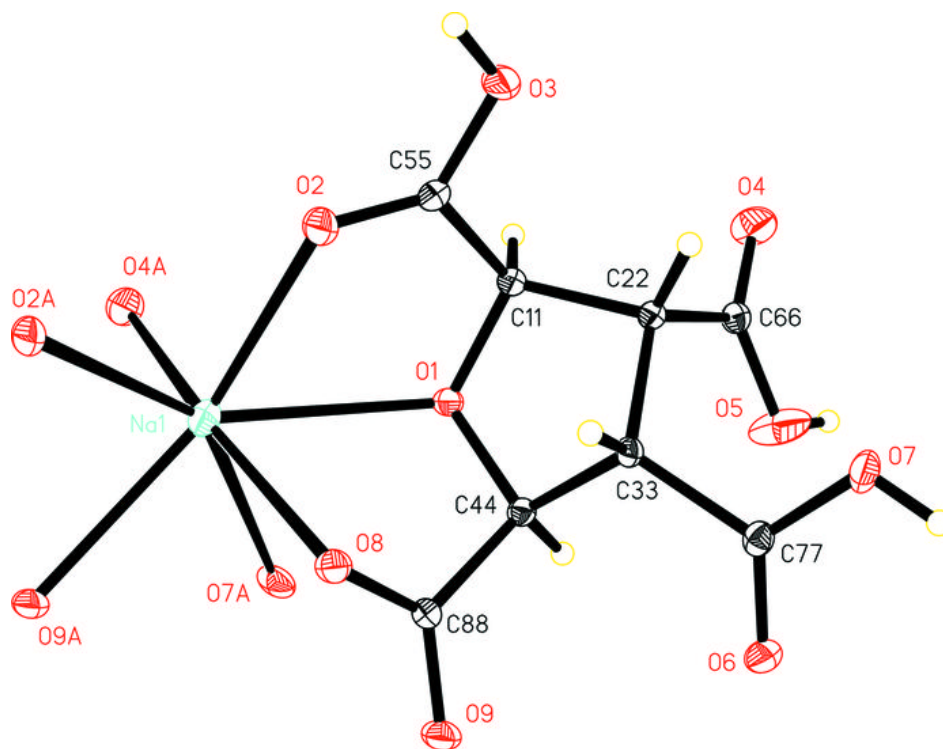


Fig. 2

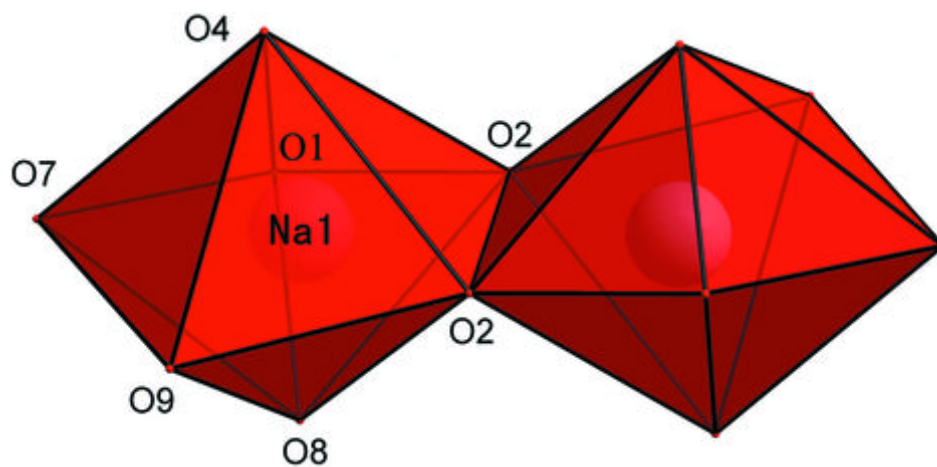


Fig. 3

