

Poly[μ_2 -aqua-diaqua(μ_8 -3-nitrobenzene-1,2-dicarboxylato)(μ_6 -3-nitrobenzene-1,2-dicarboxylato)tetrasodium]

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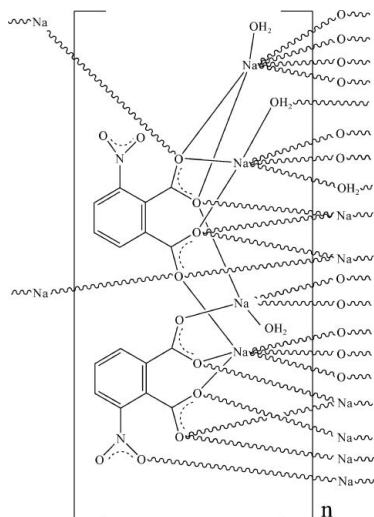
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; disorder in main residue; R factor = 0.065; wR factor = 0.208; data-to-parameter ratio = 10.2.

In the title layered coordination polymer, $[\text{Na}_4(\text{C}_8\text{H}_3\text{NO}_6)_2(\text{H}_2\text{O})_3]_n$, the doubly deprotonated 3-nitrobenzene-1,2-dicarboxylate ligands exhibit μ_8 - and μ_6 -coordination modes to the sodium ions, generating sheets lying parallel to (001). The coordination environments of the sodium ions are distorted octahedral, distorted trigonal-bipyramidal and monocapped trigonal-prismatic. One of the nitro groups is disordered over two sets of sites with site-occupancy factors 0.580 (8):0.419 (2). A network of $\text{O}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds helps to establish the packing.

Related literature

For a related structure containing the same components, see: Guo (2004).



Experimental

Crystal data

$[\text{Na}_4(\text{C}_8\text{H}_3\text{NO}_6)_2(\text{H}_2\text{O})_3]$	$\gamma = 89.371$ (2) $^\circ$
$M_r = 564.24$	$V = 1018.8$ (2) Å ³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.6871$ (8) Å	Mo $K\alpha$ radiation
$b = 10.6193$ (15) Å	$\mu = 0.23$ mm ⁻¹
$c = 14.582$ (2) Å	$T = 298$ K
$\alpha = 82.065$ (1) $^\circ$	$0.44 \times 0.38 \times 0.17$ mm
$\beta = 83.428$ (1) $^\circ$	

Data collection

Bruker SMART CCD diffractometer	5276 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2002)	3514 independent reflections
$T_{\min} = 0.905$, $T_{\max} = 0.962$	2272 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$	162 restraints
$wR(F^2) = 0.208$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.76$ e Å ⁻³
3514 reflections	$\Delta\rho_{\text{min}} = -0.69$ e Å ⁻³
344 parameters	

Table 1

Selected bond lengths (Å).

Na1—O2	2.323 (4)	Na3—O7	2.270 (4)
Na1—O8 ⁱ	2.354 (4)	Na3—O3	2.358 (4)
Na1—O2 ⁱⁱ	2.357 (4)	Na3—O4 ^v	2.390 (4)
Na1—O8	2.374 (4)	Na3—O14	2.419 (7)
Na1—O10	2.502 (5)	Na3—O11 ^{vi}	2.488 (5)
Na1—O9 ^j	2.511 (5)	Na4—O10 ^{vi}	2.363 (5)
Na2—O1 ⁱⁱⁱ	2.312 (4)	Na4—O1 ⁱⁱⁱ	2.437 (4)
Na2—O1	2.320 (4)	Na4—O15	2.448 (6)
Na2—O13	2.326 (4)	Na4—O3	2.546 (4)
Na2—O13 ^{iv}	2.353 (4)	Na4—O2 ⁱⁱⁱ	2.563 (4)
Na2—O3 ⁱⁱⁱ	2.474 (4)	Na4—O4	2.647 (4)
Na2—O4	2.475 (4)	Na4—O9 ^{vii}	2.755 (6)

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

Table 2

Hydrogen-bond geometry (Å, $^\circ$).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O13—H13A \cdots O7 ⁱⁱⁱ	0.85	1.94	2.789 (6)	180
O13—H13A \cdots O8 ⁱⁱⁱ	0.85	2.51	3.049 (5)	123
O13—H13B \cdots O10 ^{vii}	0.85	2.13	2.980 (6)	180
O13—H13B \cdots O9 ^{vii}	0.85	2.63	3.186 (6)	125
O14—H14B \cdots O6 ^{viii}	0.85	1.93	2.782 (13)	179
O14—H14B \cdots O6 ^{viii}	0.85	1.97	2.722 (15)	147
O14—H14C \cdots O6 ^{iv}	0.85	1.45	2.29 (2)	166
O14—H14C \cdots N1 ^v	0.85	2.27	3.078 (10)	160
O14—H14C \cdots O6 ^v	0.85	2.55	3.388 (18)	171
O15—H15B \cdots O5	0.85	2.13	2.951 (7)	162
O15—H15C \cdots O9 ^{vii}	0.85	2.29	2.782 (8)	117
O15—H15C \cdots O11 ^{vii}	0.85	2.48	3.302 (8)	163

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5692).

References

- Bruker (2002). *SADABS*, *SAINTE* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Guo, M.-L. (2004). *Acta Cryst.* **E60**, m1684–m1685.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2010). E66, m1509-m1510 [doi:10.1107/S1600536810044600]

Poly[μ_2 -aqua-diaqua(μ_8 -3-nitrobenzene-1,2-dicarboxylato)(μ_6 -3-nitrobenzene-1,2-dicarboxylato)tetrasodium]

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Comment

Ming Ling Guo has obtained one kind of sodium complex $\{[\text{Na}(\text{C}_8\text{H}_4\text{NO}_6)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}\}_n$ (Guo, 2004) based on 3-Nitrobenzene-1,2-dicarboxylic acid ligand.

Here we report another kind of sodium complex (I) based on the same ligand. Different from $\{[\text{Na}(\text{C}_8\text{H}_4\text{NO}_6)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}\}_n$, the formula for the title complex is $[\text{Na}_4(\text{C}_8\text{H}_3\text{NO}_6)_2(\text{H}_2\text{O})_3]_n$. X-ray single-crystal diffraction analysis indicates the presence of four independent Na^{I} ions, two 3-Nitrobenzene-1,2-dicarboxylato and three coordinated water molecules in the asymmetric unit. Only one independent Na^{I} ion can be found in complex of $\{[\text{Na}(\text{C}_8\text{H}_4\text{NO}_6)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}\}_n$. Moreover, the ligand in the title complex is completely deprotonated, which is different from the uncomplete form in complex of $\{[\text{Na}(\text{C}_8\text{H}_4\text{NO}_6)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}\}_n$.

In the title complex, the coordination geometry (Fig. 1) around $\text{Na}1^{\text{I}}$ and $\text{Na}2^{\text{I}}$ ions could be described as distorted octahedral arrangements with coordination number of 6, all the coordinated atoms are oxygen atoms. For $\text{Na}1^{\text{I}}$ center, all the six oxygen atoms come from carboxylate groups. For $\text{Na}2^{\text{I}}$ center, four oxygen atoms come from carboxylate groups, two of them come from coordinated water molecules. The coordination numbers of $\text{Na}3^{\text{I}}$ and $\text{Na}4^{\text{I}}$ ions are 5 and 7, and the geometries around them could be described as distorted trigonal bipyramid and monocapped octahedron prism arrangements. Around $\text{Na}3^{\text{I}}$ center, three oxygen atoms come from carboxylate groups, one is nitro oxygen atom, and the last comes from coordinated water molecule. Concerning $\text{Na}4^{\text{I}}$ center, six of the oxygen atoms belong to carboxylate groups and only one oxygen atom is from coordinated water molecule. 3-Nitrobenzene-1,2-dicarboxylic acid ligands exhibit two coordination modes (Fig. 2), which can be classified as μ_8 -(κ^{12} , O^1 : O^1 : O^1 : O^2 : O^2 : O^2 : O^3 : O^3 : O^3 : O^4 : O^4 : O^4) and μ_6 -(κ^8 , O^7 : O^8 : O^8 : O^9 : O^9 : O^{10} : O^{10} : O^{11}), and a two-dimensional (Fig. 3) polymeric framework can be assembled by oxygen atoms of the ligands through the two coordination modes. We can clearly see that it is different from the complex synthesized by Ming Ling Guo, which forms polymeric chains by way of edge-sharing *via* pairs of water molecules between $\text{NaO}(\text{H}_2\text{O})_5$ octahedra.

Experimental

A mixture of strontium chloride hexahydrate (0.0267 g, 0.1 mmol), sodium hydroxide (0.0080 g, 0.2 mmol), 3-Nitrobenzene-1,2-dicarboxylic acid (0.0211 g, 0.1 mmol), and H_2O (20 mL) was placed in a Parr Teflon-lined stainless steel vessel (25 ml), and then the vessel was sealed and heated at 443.15 K for 4 days. Then the vessel was cooled to 373.15 K at a rate of 5 K h^{-1} and slowly cooled to room temperature. Colorless, block single crystals suitable for X-ray diffraction were obtained.

Figures

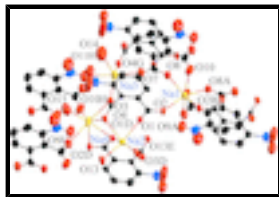


Fig. 1. Coordination environment of Na^{I} ions in the title complex. Non-hydrogen atoms are shown as 30% probability ellipsoids. Hydrogen atoms are omitted for clarity. Symmetry codes: (A) $-x, -y, -z + 1$; (B) $-x + 1, -y, -z + 1$; (D) $-x + 1, -y + 1, -z + 1$; (E) $-x + 2, -y + 1, -z + 1$; (G) $x - 1, y, z$; (H) $x, y + 1, z$; (I) $x + 1, y + 1, z$.

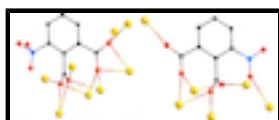


Fig. 2. Coordination modes of 3-Nitrobenzene-1,2-dicarboxylic acid ligands in the title complex. Hydrogen atoms are omitted for clarity.

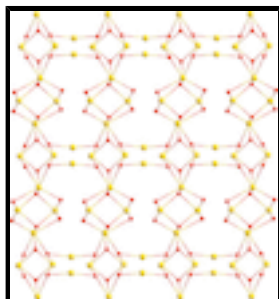


Fig. 3. View of two-dimensional framework along c axis in the title complex.

Poly[μ_2 -aqua-diaqua(μ_8 -3-nitrobenzene-1,2-dicarboxylato)(μ_6 -3-nitrobenzene-1,2-dicarboxylato)tetrasodium]

Crystal data

$[\text{Na}_4(\text{C}_8\text{H}_3\text{NO}_6)_2(\text{H}_2\text{O})_3]$

$M_r = 564.24$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 6.6871$ (8) Å

$b = 10.6193$ (15) Å

$c = 14.582$ (2) Å

$\alpha = 82.065$ (1)°

$\beta = 83.428$ (1)°

$\gamma = 89.371$ (2)°

$V = 1018.8$ (2) Å³

$Z = 2$

$F(000) = 572$

$D_x = 1.839$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1631 reflections

$\theta = 2.8\text{--}28.2^\circ$

$\mu = 0.23$ mm⁻¹

$T = 298$ K

Block, colourless

$0.44 \times 0.38 \times 0.17$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

phi and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2002)

$T_{\text{min}} = 0.905$, $T_{\text{max}} = 0.962$

3514 independent reflections

2272 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.8^\circ$

$h = -7 \rightarrow 7$

$k = -11 \rightarrow 12$

5276 measured reflections

$l = -17 \rightarrow 14$

Refinement

Refinement on F^2

Primary atom site location: structure-invariant direct methods

Least-squares matrix: full

Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.065$

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.208$

H-atom parameters constrained

$S = 1.04$

$w = 1/[\sigma^2(F_o^2) + (0.1154P)^2 + 0.9421P]$

where $P = (F_o^2 + 2F_c^2)/3$

3514 reflections

$(\Delta/\sigma)_{\max} < 0.001$

344 parameters

$\Delta\rho_{\max} = 0.76 \text{ e } \text{\AA}^{-3}$

162 restraints

$\Delta\rho_{\min} = -0.69 \text{ e } \text{\AA}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Na1	0.2359 (3)	0.00241 (17)	0.50423 (14)	0.0330 (5)	
Na2	0.7386 (3)	0.50445 (17)	0.49820 (13)	0.0302 (5)	
Na3	0.0578 (3)	0.5315 (2)	0.28461 (17)	0.0479 (6)	
Na4	0.5016 (4)	0.7672 (2)	0.37516 (15)	0.0522 (7)	
N1	0.6311 (9)	0.5233 (5)	0.1143 (3)	0.0533 (14)	
N2	0.1544 (9)	-0.1646 (5)	0.1478 (4)	0.0556 (14)	
O1	0.5149 (5)	0.3531 (3)	0.4692 (2)	0.0299 (8)	
O2	0.5038 (5)	0.1449 (3)	0.4694 (2)	0.0335 (8)	
O3	0.3758 (5)	0.5587 (3)	0.3345 (2)	0.0328 (8)	
O4	0.7092 (5)	0.5666 (3)	0.3304 (2)	0.0324 (8)	
O5	0.5745 (9)	0.6168 (4)	0.1406 (3)	0.0703 (13)	
O6	0.645 (2)	0.5248 (9)	0.0290 (6)	0.084 (3)	0.581 (14)
O6'	0.794 (3)	0.5271 (13)	0.0538 (10)	0.085 (4)	0.419 (14)
O7	0.0464 (6)	0.3179 (4)	0.3283 (3)	0.0511 (10)	
O8	0.0225 (6)	0.1255 (3)	0.4077 (3)	0.0434 (9)	
O9	-0.1331 (8)	-0.1086 (4)	0.3542 (3)	0.0583 (11)	
O10	0.1955 (8)	-0.1197 (4)	0.3725 (3)	0.0644 (11)	

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O11	0.0978 (9)	-0.2416 (5)	0.2146 (3)	0.0735 (13)
O12	0.2147 (11)	-0.1972 (6)	0.0737 (4)	0.104 (2)
O13	1.0048 (6)	0.6475 (3)	0.4843 (3)	0.0438 (10)
H13A	0.9897	0.6583	0.5414	0.053*
H13B	1.0595	0.7139	0.4526	0.053*
O14	0.0893 (12)	0.5436 (6)	0.1167 (5)	0.116 (2)
H14B	0.1713	0.5230	0.0724	0.139*
H14C	-0.0290	0.5412	0.1009	0.139*
O15	0.6276 (9)	0.8569 (5)	0.2156 (4)	0.0861 (16)
H15B	0.5852	0.7932	0.1939	0.103*
H15C	0.7525	0.8465	0.2201	0.103*
C1	0.5209 (7)	0.2562 (4)	0.4276 (3)	0.0250 (10)
C2	0.5488 (7)	0.5173 (4)	0.3145 (3)	0.0235 (10)
C3	0.5542 (7)	0.2752 (4)	0.3225 (3)	0.0256 (11)
C4	0.5689 (7)	0.3952 (4)	0.2700 (3)	0.0224 (10)
C5	0.6096 (8)	0.4004 (5)	0.1743 (3)	0.0328 (12)
C6	0.6326 (9)	0.2933 (5)	0.1290 (4)	0.0418 (14)
H6	0.6595	0.3009	0.0645	0.050*
C7	0.6145 (8)	0.1769 (5)	0.1820 (4)	0.0384 (13)
H7	0.6276	0.1036	0.1535	0.046*
C8	0.5770 (8)	0.1676 (5)	0.2771 (4)	0.0320 (12)
H8	0.5664	0.0875	0.3124	0.038*
C9	0.0500 (8)	0.1990 (5)	0.3334 (4)	0.0368 (13)
C10	0.0487 (11)	-0.0812 (5)	0.3305 (4)	0.0437 (15)
C11	0.0931 (8)	0.1445 (5)	0.2430 (4)	0.0379 (13)
C12	0.0976 (8)	0.0117 (5)	0.2412 (4)	0.0376 (13)
C13	0.1464 (9)	-0.0290 (6)	0.1550 (4)	0.0448 (14)
C14	0.1874 (10)	0.0541 (7)	0.0734 (4)	0.0580 (18)
H14A	0.2219	0.0232	0.0171	0.070*
C15	0.1767 (11)	0.1815 (7)	0.0762 (5)	0.0615 (19)
H15A	0.2003	0.2383	0.0215	0.074*
C16	0.1308 (9)	0.2257 (6)	0.1606 (5)	0.0510 (16)
H16	0.1252	0.3129	0.1621	0.061*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0312 (11)	0.0249 (11)	0.0412 (12)	-0.0031 (8)	-0.0024 (9)	0.0004 (9)
Na2	0.0293 (11)	0.0276 (11)	0.0333 (11)	-0.0011 (8)	-0.0008 (8)	-0.0051 (8)
Na3	0.0333 (13)	0.0374 (13)	0.0668 (16)	-0.0031 (10)	-0.0023 (11)	0.0127 (11)
Na4	0.0914 (19)	0.0293 (12)	0.0323 (12)	0.0099 (12)	0.0022 (12)	0.0002 (9)
N1	0.092 (4)	0.037 (3)	0.026 (3)	-0.010 (3)	0.010 (3)	-0.001 (2)
N2	0.070 (4)	0.059 (4)	0.038 (3)	0.009 (3)	-0.004 (3)	-0.008 (3)
O1	0.0361 (18)	0.0217 (16)	0.0308 (16)	-0.0013 (13)	-0.0004 (14)	-0.0032 (13)
O2	0.0379 (19)	0.0186 (16)	0.0406 (19)	-0.0014 (14)	-0.0009 (15)	0.0050 (14)
O3	0.0352 (18)	0.0261 (16)	0.0368 (18)	0.0034 (14)	-0.0020 (14)	-0.0061 (14)
O4	0.0358 (18)	0.0217 (16)	0.0388 (18)	-0.0050 (14)	0.0000 (14)	-0.0046 (13)
O5	0.108 (3)	0.043 (2)	0.052 (2)	0.008 (2)	0.010 (2)	0.004 (2)

O6	0.135 (7)	0.063 (5)	0.047 (5)	-0.008 (5)	0.009 (5)	-0.002 (4)
O6'	0.110 (8)	0.061 (6)	0.065 (6)	0.008 (6)	0.040 (6)	0.014 (5)
O7	0.052 (2)	0.034 (2)	0.064 (2)	0.0012 (17)	-0.0030 (19)	0.0017 (17)
O8	0.052 (2)	0.0327 (18)	0.0437 (19)	-0.0015 (16)	-0.0045 (16)	0.0015 (16)
O9	0.082 (3)	0.041 (2)	0.048 (2)	-0.010 (2)	0.006 (2)	-0.0049 (17)
O10	0.090 (3)	0.051 (2)	0.049 (2)	0.024 (2)	-0.009 (2)	-0.0013 (18)
O11	0.114 (3)	0.049 (2)	0.053 (2)	0.011 (2)	0.008 (2)	-0.004 (2)
O12	0.171 (5)	0.081 (4)	0.057 (3)	0.001 (4)	0.020 (3)	-0.025 (3)
O13	0.042 (2)	0.0249 (18)	0.062 (2)	-0.0068 (16)	0.0022 (18)	-0.0012 (17)
O14	0.143 (5)	0.094 (4)	0.099 (4)	0.000 (4)	0.020 (4)	-0.002 (3)
O15	0.088 (4)	0.057 (3)	0.111 (4)	-0.003 (3)	-0.002 (3)	-0.008 (3)
C1	0.020 (2)	0.024 (3)	0.031 (3)	0.0011 (19)	-0.0023 (19)	-0.002 (2)
C2	0.029 (3)	0.015 (2)	0.025 (2)	-0.004 (2)	0.002 (2)	-0.0002 (18)
C3	0.019 (2)	0.028 (3)	0.030 (3)	-0.001 (2)	-0.0028 (19)	-0.004 (2)
C4	0.021 (2)	0.022 (2)	0.024 (2)	-0.0027 (19)	0.0006 (19)	-0.0054 (19)
C5	0.038 (3)	0.029 (3)	0.030 (3)	-0.004 (2)	0.001 (2)	-0.003 (2)
C6	0.056 (4)	0.042 (3)	0.028 (3)	0.001 (3)	0.002 (3)	-0.011 (2)
C7	0.043 (3)	0.036 (3)	0.039 (3)	-0.002 (2)	-0.003 (3)	-0.018 (3)
C8	0.031 (3)	0.023 (3)	0.044 (3)	0.000 (2)	-0.007 (2)	-0.011 (2)
C9	0.024 (3)	0.030 (3)	0.057 (4)	-0.004 (2)	-0.006 (2)	-0.003 (3)
C10	0.069 (4)	0.029 (3)	0.033 (3)	0.014 (3)	-0.008 (3)	-0.004 (2)
C11	0.026 (3)	0.035 (3)	0.048 (3)	-0.004 (2)	-0.003 (2)	0.009 (2)
C12	0.031 (3)	0.039 (3)	0.041 (3)	0.001 (2)	-0.005 (2)	0.003 (2)
C13	0.047 (4)	0.047 (4)	0.039 (3)	0.000 (3)	-0.005 (3)	0.001 (3)
C14	0.061 (4)	0.075 (5)	0.035 (4)	-0.008 (4)	-0.005 (3)	0.004 (3)
C15	0.064 (5)	0.063 (5)	0.050 (4)	-0.018 (4)	-0.007 (3)	0.020 (3)
C16	0.044 (4)	0.045 (4)	0.059 (4)	-0.010 (3)	-0.007 (3)	0.011 (3)

Geometric parameters (Å, °)

Na1—O2	2.323 (4)	O4—Na3 ^{viii}	2.390 (4)
Na1—O8 ⁱ	2.354 (4)	O7—C9	1.255 (6)
Na1—O2 ⁱⁱ	2.357 (4)	O8—C9	1.240 (7)
Na1—O8	2.374 (4)	O8—Na1 ⁱ	2.354 (4)
Na1—O10	2.502 (5)	O9—C10	1.252 (8)
Na1—O9 ⁱ	2.511 (5)	O9—Na1 ⁱ	2.511 (5)
Na2—O1 ⁱⁱⁱ	2.312 (4)	O9—Na4 ^{ix}	2.755 (6)
Na2—O1	2.320 (4)	O10—C10	1.251 (7)
Na2—O13	2.326 (4)	O10—Na4 ^x	2.363 (5)
Na2—O13 ^{iv}	2.353 (4)	O11—Na3 ^x	2.488 (5)
Na2—O3 ⁱⁱⁱ	2.474 (4)	O13—Na2 ^{iv}	2.353 (4)
Na2—O4	2.475 (4)	O13—H13A	0.8498
Na3—O7	2.270 (4)	O13—H13B	0.8500
Na3—O3	2.358 (4)	O14—H14B	0.8500
Na3—O4 ^v	2.390 (4)	O14—H14C	0.8500
Na3—O14	2.419 (7)	O15—H15B	0.8500
Na3—O11 ^{vi}	2.488 (5)	O15—H15C	0.8500

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Na4—O10 ^{vi}	2.363 (5)	C1—C3	1.509 (7)
Na4—O1 ⁱⁱⁱ	2.437 (4)	C2—C4	1.526 (6)
Na4—O15	2.448 (6)	C3—C4	1.391 (7)
Na4—O3	2.546 (4)	C3—C8	1.396 (7)
Na4—O2 ⁱⁱⁱ	2.563 (4)	C4—C5	1.385 (7)
Na4—O4	2.647 (4)	C5—C6	1.390 (7)
Na4—O9 ^{vii}	2.755 (6)	C6—C7	1.363 (8)
N1—O5	1.156 (6)	C6—H6	0.9300
N1—O6	1.236 (10)	C7—C8	1.371 (7)
N1—O6'	1.320 (15)	C7—H7	0.9300
N1—C5	1.466 (7)	C8—H8	0.9300
N2—O12	1.204 (7)	C9—C11	1.509 (8)
N2—O11	1.209 (7)	C10—C12	1.527 (8)
N2—C13	1.458 (8)	C11—C16	1.378 (8)
O1—C1	1.263 (6)	C11—C12	1.414 (8)
O1—Na2 ⁱⁱⁱ	2.312 (4)	C12—C13	1.387 (8)
O1—Na4 ⁱⁱⁱ	2.437 (4)	C13—C14	1.382 (8)
O2—C1	1.253 (6)	C14—C15	1.359 (10)
O2—Na1 ⁱⁱ	2.357 (4)	C14—H14A	0.9300
O2—Na4 ⁱⁱⁱ	2.563 (4)	C15—C16	1.379 (10)
O3—C2	1.250 (6)	C15—H15A	0.9300
O3—Na2 ⁱⁱⁱ	2.474 (4)	C16—H16	0.9300
O4—C2	1.257 (6)		
O2—Na1—O8 ⁱ	159.75 (16)	C2—O4—Na2	106.5 (3)
O2—Na1—O2 ⁱⁱ	82.43 (13)	Na3 ^{viii} —O4—Na2	93.63 (14)
O8 ⁱ —Na1—O2 ⁱⁱ	95.74 (14)	C2—O4—Na4	89.2 (3)
O2—Na1—O8	94.71 (14)	Na3 ^{viii} —O4—Na4	133.50 (16)
O8 ⁱ —Na1—O8	95.73 (14)	Na2—O4—Na4	88.06 (12)
O2 ⁱⁱ —Na1—O8	153.10 (16)	C9—O7—Na3	167.2 (4)
O2—Na1—O10	112.15 (17)	C9—O8—Na1 ⁱ	139.0 (4)
O8 ⁱ —Na1—O10	87.46 (16)	C9—O8—Na1	134.7 (4)
O2 ⁱⁱ —Na1—O10	82.65 (15)	Na1 ⁱ —O8—Na1	84.27 (14)
O8—Na1—O10	73.64 (15)	C10—O9—Na1 ⁱ	105.9 (4)
O2—Na1—O9 ⁱ	87.49 (15)	C10—O9—Na4 ^{ix}	161.6 (4)
O8 ⁱ —Na1—O9 ⁱ	74.31 (15)	Na1 ⁱ —O9—Na4 ^{ix}	91.05 (16)
O2 ⁱⁱ —Na1—O9 ⁱ	111.21 (16)	C10—O10—Na4 ^x	149.5 (4)
O8—Na1—O9 ⁱ	95.30 (16)	C10—O10—Na1	111.7 (4)
O10—Na1—O9 ⁱ	157.80 (19)	Na4 ^x —O10—Na1	97.44 (19)
O1 ⁱⁱⁱ —Na2—O1	93.04 (13)	N2—O11—Na3 ^x	148.5 (4)
O1 ⁱⁱⁱ —Na2—O13	96.65 (14)	Na2—O13—Na2 ^{iv}	96.95 (14)
O1—Na2—O13	163.33 (16)	Na2—O13—H13A	94.9
O1 ⁱⁱⁱ —Na2—O13 ^{iv}	162.13 (16)	Na2 ^{iv} —O13—H13A	94.7
O1—Na2—O13 ^{iv}	91.84 (14)	Na2—O13—H13B	144.8

O13—Na2—O13 ^{iv}	83.05 (14)	Na2 ^{iv} —O13—H13B	106.7
O1 ⁱⁱⁱ —Na2—O3 ⁱⁱⁱ	75.90 (13)	H13A—O13—H13B	108.4
O1—Na2—O3 ⁱⁱⁱ	86.70 (13)	Na3—O14—H14B	139.6
O13—Na2—O3 ⁱⁱⁱ	108.79 (15)	Na3—O14—H14C	107.4
O13 ^{iv} —Na2—O3 ⁱⁱⁱ	87.24 (14)	H14B—O14—H14C	108.1
O1 ⁱⁱⁱ —Na2—O4	88.52 (13)	Na4—O15—H15B	91.7
O1—Na2—O4	78.00 (12)	Na4—O15—H15C	97.8
O13—Na2—O4	88.70 (14)	H15B—O15—H15C	107.9
O13 ^{iv} —Na2—O4	109.32 (14)	O2—C1—O1	123.1 (4)
O3 ⁱⁱⁱ —Na2—O4	157.58 (14)	O2—C1—C3	118.4 (4)
O7—Na3—O3	94.91 (15)	O1—C1—C3	118.4 (4)
O7—Na3—O4 ^v	95.67 (15)	O2—C1—Na4 ⁱⁱⁱ	64.5 (3)
O3—Na3—O4 ^v	140.24 (16)	O1—C1—Na4 ⁱⁱⁱ	58.8 (2)
O7—Na3—O14	101.1 (2)	C3—C1—Na4 ⁱⁱⁱ	174.0 (3)
O3—Na3—O14	110.0 (2)	O3—C2—O4	125.2 (4)
O4 ^v —Na3—O14	105.3 (2)	O3—C2—C4	118.1 (4)
O7—Na3—O11 ^{vi}	171.5 (2)	O4—C2—C4	116.7 (4)
O3—Na3—O11 ^{vi}	84.59 (17)	O3—C2—Na4	60.6 (2)
O4 ^v —Na3—O11 ^{vi}	90.07 (16)	O4—C2—Na4	65.3 (2)
O14—Na3—O11 ^{vi}	71.2 (2)	C4—C2—Na4	172.4 (3)
O10 ^{vi} —Na4—O1 ⁱⁱⁱ	105.27 (16)	O3—C2—Na2	103.5 (3)
O10 ^{vi} —Na4—O15	93.48 (18)	O4—C2—Na2	50.4 (2)
O1 ⁱⁱⁱ —Na4—O15	161.21 (19)	C4—C2—Na2	114.3 (3)
O10 ^{vi} —Na4—O3	97.84 (18)	Na4—C2—Na2	72.89 (12)
O1 ⁱⁱⁱ —Na4—O3	82.68 (13)	C4—C3—C8	119.3 (4)
O15—Na4—O3	96.15 (17)	C4—C3—C1	122.5 (4)
O10 ^{vi} —Na4—O2 ⁱⁱⁱ	81.22 (15)	C8—C3—C1	118.2 (4)
O1 ⁱⁱⁱ —Na4—O2 ⁱⁱⁱ	52.44 (11)	C5—C4—C3	117.1 (4)
O15—Na4—O2 ⁱⁱⁱ	131.77 (18)	C5—C4—C2	120.5 (4)
O3—Na4—O2 ⁱⁱⁱ	132.08 (14)	C3—C4—C2	122.4 (4)
O10 ^{vi} —Na4—O4	147.23 (18)	C4—C5—C6	123.7 (5)
O1 ⁱⁱⁱ —Na4—O4	82.10 (12)	C4—C5—N1	120.4 (4)
O15—Na4—O4	82.72 (16)	C6—C5—N1	116.0 (5)
O3—Na4—O4	50.71 (12)	C7—C6—C5	118.0 (5)
O2 ⁱⁱⁱ —Na4—O4	124.99 (14)	C7—C6—H6	121.0
O10 ^{vi} —Na4—O9 ^{vii}	121.48 (18)	C5—C6—H6	121.0
O1 ⁱⁱⁱ —Na4—O9 ^{vii}	103.59 (15)	C6—C7—C8	120.2 (5)
O15—Na4—O9 ^{vii}	64.33 (17)	C6—C7—H7	119.9
O3—Na4—O9 ^{vii}	135.66 (16)	C8—C7—H7	119.9
O2 ⁱⁱⁱ —Na4—O9 ^{vii}	77.88 (14)	C7—C8—C3	121.7 (5)
O4—Na4—O9 ^{vii}	86.19 (14)	C7—C8—H8	119.1
O5—N1—O6	115.1 (7)	C3—C8—H8	119.1

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O5—N1—O6'	118.7 (8)	O8—C9—O7	123.9 (5)
O6—N1—O6'	51.1 (8)	O8—C9—C11	119.2 (5)
O5—N1—C5	121.9 (5)	O7—C9—C11	116.9 (5)
O6—N1—C5	118.6 (6)	O10—C10—O9	127.4 (6)
O6'—N1—C5	111.5 (7)	O10—C10—C12	116.0 (6)
O12—N2—O11	121.3 (6)	O9—C10—C12	116.6 (5)
O12—N2—C13	118.4 (6)	O10—C10—Na1 ⁱ	99.6 (4)
O11—N2—C13	120.3 (5)	O9—C10—Na1 ⁱ	51.2 (3)
C1—O1—Na2 ⁱⁱⁱ	132.4 (3)	C12—C10—Na1 ⁱ	120.5 (3)
C1—O1—Na2	137.6 (3)	C16—C11—C12	119.3 (6)
Na2 ⁱⁱⁱ —O1—Na2	86.96 (13)	C16—C11—C9	119.4 (5)
C1—O1—Na4 ⁱⁱⁱ	94.9 (3)	C12—C11—C9	121.3 (5)
Na2 ⁱⁱⁱ —O1—Na4 ⁱⁱⁱ	97.15 (14)	C13—C12—C11	116.9 (5)
Na2—O1—Na4 ⁱⁱⁱ	94.76 (14)	C13—C12—C10	122.3 (5)
C1—O2—Na1	133.0 (3)	C11—C12—C10	120.8 (5)
C1—O2—Na1 ⁱⁱ	127.2 (3)	C14—C13—C12	122.8 (6)
Na1—O2—Na1 ⁱⁱ	97.57 (13)	C14—C13—N2	117.2 (6)
C1—O2—Na4 ⁱⁱⁱ	89.3 (3)	C12—C13—N2	119.9 (5)
Na1—O2—Na4 ⁱⁱⁱ	100.56 (15)	C15—C14—C13	119.4 (6)
Na1 ⁱⁱ —O2—Na4 ⁱⁱⁱ	95.93 (14)	C15—C14—H14A	120.3
C2—O3—Na3	135.0 (3)	C13—C14—H14A	120.3
C2—O3—Na2 ⁱⁱⁱ	110.4 (3)	C14—C15—C16	119.5 (6)
Na3—O3—Na2 ⁱⁱⁱ	94.88 (14)	C14—C15—H15A	120.3
C2—O3—Na4	94.0 (3)	C16—C15—H15A	120.3
Na3—O3—Na4	124.18 (16)	C11—C16—C15	122.0 (6)
Na2 ⁱⁱⁱ —O3—Na4	88.46 (12)	C11—C16—H16	119.0
C2—O4—Na3 ^{viii}	133.8 (3)	C15—C16—H16	119.0
O1 ⁱⁱⁱ —Na2—O1—C1	160.4 (5)	Na1 ⁱⁱ —O2—C1—C3	77.2 (5)
O13—Na2—O1—C1	34.9 (8)	Na4 ⁱⁱⁱ —O2—C1—C3	174.1 (4)
O13 ^{iv} —Na2—O1—C1	-36.8 (5)	Na2 ⁱⁱⁱ —O1—C1—O2	-99.4 (5)
O3 ⁱⁱⁱ —Na2—O1—C1	-123.9 (5)	Na2—O1—C1—O2	107.5 (5)
O4—Na2—O1—C1	72.6 (5)	Na4 ⁱⁱⁱ —O1—C1—O2	5.0 (5)
C2—Na2—O1—C1	81.8 (5)	Na2 ⁱⁱⁱ —O1—C1—C3	81.9 (5)
O1 ⁱⁱⁱ —Na2—O1—Na2 ⁱⁱⁱ	0.0	Na2—O1—C1—C3	-71.3 (6)
O13—Na2—O1—Na2 ⁱⁱⁱ	-125.5 (5)	Na4 ⁱⁱⁱ —O1—C1—C3	-173.8 (4)
O13 ^{iv} —Na2—O1—Na2 ⁱⁱⁱ	162.80 (15)	Na3—O3—C2—O4	160.6 (3)
O3 ⁱⁱⁱ —Na2—O1—Na2 ⁱⁱⁱ	75.68 (12)	Na2 ⁱⁱⁱ —O3—C2—O4	-79.4 (5)
O4—Na2—O1—Na2 ⁱⁱⁱ	-87.84 (12)	Na4—O3—C2—O4	10.4 (5)
C2—Na2—O1—Na2 ⁱⁱⁱ	-78.63 (14)	Na3—O3—C2—C4	-21.3 (6)
O1 ⁱⁱⁱ —Na2—O1—Na4 ⁱⁱⁱ	-96.93 (14)	Na2 ⁱⁱⁱ —O3—C2—C4	98.7 (4)
O13—Na2—O1—Na4 ⁱⁱⁱ	137.5 (5)	Na4—O3—C2—C4	-171.5 (4)
O13 ^{iv} —Na2—O1—Na4 ⁱⁱⁱ	65.87 (15)	Na3 ^{viii} —O4—C2—O3	-170.1 (3)

O3 ⁱⁱⁱ —Na2—O1—Na4 ⁱⁱⁱ	-21.25 (13)	Na2—O4—C2—O3	77.8 (5)
O4—Na2—O1—Na4 ⁱⁱⁱ	175.23 (14)	Na4—O4—C2—O3	-10.0 (5)
C2—Na2—O1—Na4 ⁱⁱⁱ	-175.56 (18)	Na3 ^{viii} —O4—C2—C4	11.7 (6)
O8 ⁱ —Na1—O2—C1	-110.5 (6)	Na2—O4—C2—C4	-100.4 (4)
O2 ⁱⁱ —Na1—O2—C1	163.4 (5)	Na4—O4—C2—C4	171.9 (4)
O8—Na1—O2—C1	10.3 (5)	O10 ^{vi} —Na4—C2—O3	-18.2 (3)
O10—Na1—O2—C1	84.6 (5)	O1 ⁱⁱⁱ —Na4—C2—O3	86.0 (3)
O9 ⁱ —Na1—O2—C1	-84.8 (5)	O15—Na4—C2—O3	-110.6 (3)
O8 ⁱ —Na1—O2—Na1 ⁱⁱ	86.1 (4)	O2 ⁱⁱⁱ —Na4—C2—O3	94.5 (3)
O2 ⁱⁱ —Na1—O2—Na1 ⁱⁱ	0.0	O4—Na4—C2—O3	170.6 (4)
O8—Na1—O2—Na1 ⁱⁱ	-153.09 (16)	O9 ^{vii} —Na4—C2—O3	-171.8 (3)
O10—Na1—O2—Na1 ⁱⁱ	-78.85 (17)	C1 ⁱⁱⁱ —Na4—C2—O3	88.3 (3)
O9 ⁱ —Na1—O2—Na1 ⁱⁱ	111.79 (16)	O10 ^{vi} —Na4—C2—O4	171.2 (3)
C10 ⁱ —Na1—O2—Na1 ⁱⁱ	119.93 (16)	O1 ⁱⁱⁱ —Na4—C2—O4	-84.6 (3)
O8 ⁱ —Na1—O2—Na4 ⁱⁱⁱ	-11.5 (5)	O15—Na4—C2—O4	78.8 (3)
O2 ⁱⁱ —Na1—O2—Na4 ⁱⁱⁱ	-97.52 (16)	O3—Na4—C2—O4	-170.6 (4)
O8—Na1—O2—Na4 ⁱⁱⁱ	109.39 (15)	O2 ⁱⁱⁱ —Na4—C2—O4	-76.2 (3)
O10—Na1—O2—Na4 ⁱⁱⁱ	-176.37 (14)	O9 ^{vii} —Na4—C2—O4	17.6 (3)
O9 ⁱ —Na1—O2—Na4 ⁱⁱⁱ	14.27 (16)	O10 ^{vi} —Na4—C2—C4	64 (2)
O7—Na3—O3—C2	59.7 (4)	O1 ⁱⁱⁱ —Na4—C2—C4	168 (2)
O4 ^v —Na3—O3—C2	164.7 (4)	O15—Na4—C2—C4	-28 (2)
O14—Na3—O3—C2	-44.0 (5)	O3—Na4—C2—C4	82 (2)
O11 ^{vi} —Na3—O3—C2	-111.7 (4)	O2 ⁱⁱⁱ —Na4—C2—C4	177 (2)
O7—Na3—O3—Na2 ⁱⁱⁱ	-65.70 (16)	O4—Na4—C2—C4	-107 (2)
O4 ^v —Na3—O3—Na2 ⁱⁱⁱ	39.3 (3)	O9 ^{vii} —Na4—C2—C4	-89 (2)
O14—Na3—O3—Na2 ⁱⁱⁱ	-169.41 (19)	O10 ^{vi} —Na4—C2—Na2	-135.11 (17)
O11 ^{vi} —Na3—O3—Na2 ⁱⁱⁱ	122.85 (15)	O1 ⁱⁱⁱ —Na4—C2—Na2	-30.95 (11)
O7—Na3—O3—Na4	-157.15 (19)	O15—Na4—C2—Na2	132.50 (17)
O4 ^v —Na3—O3—Na4	-52.2 (3)	O3—Na4—C2—Na2	-116.9 (3)
O14—Na3—O3—Na4	99.1 (2)	O2 ⁱⁱⁱ —Na4—C2—Na2	-22.5 (2)
O11 ^{vi} —Na3—O3—Na4	31.4 (2)	O4—Na4—C2—Na2	53.7 (2)
O10 ^{vi} —Na4—O3—C2	164.5 (3)	O9 ^{vii} —Na4—C2—Na2	71.27 (15)
O1 ⁱⁱⁱ —Na4—O3—C2	-91.0 (3)	O1 ⁱⁱⁱ —Na2—C2—O3	-20.1 (3)
O15—Na4—O3—C2	70.2 (3)	O1—Na2—C2—O3	78.8 (3)
O2 ⁱⁱⁱ —Na4—O3—C2	-110.2 (3)	O13—Na2—C2—O3	-114.2 (3)
O4—Na4—O3—C2	-5.2 (3)	O13 ^{iv} —Na2—C2—O3	153.7 (3)
O9 ^{vii} —Na4—O3—C2	11.0 (4)	O3 ⁱⁱⁱ —Na2—C2—O3	40.3 (4)
O10 ^{vi} —Na4—O3—Na3	9.7 (2)	O4—Na2—C2—O3	-124.8 (5)
O1 ⁱⁱⁱ —Na4—O3—Na3	114.2 (2)	O1 ⁱⁱⁱ —Na2—C2—O4	104.7 (3)
O15—Na4—O3—Na3	-84.7 (2)	O1—Na2—C2—O4	-156.4 (3)
O2 ⁱⁱⁱ —Na4—O3—Na3	94.9 (2)	O13—Na2—C2—O4	10.5 (3)

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O4—Na4—O3—Na3	-160.0 (2)	O13 ^{iv} —Na2—C2—O4	-81.5 (3)
O9 ^{vii} —Na4—O3—Na3	-143.8 (2)	O3 ⁱⁱⁱ —Na2—C2—O4	165.1 (3)
O10 ^{vi} —Na4—O3—Na2 ⁱⁱⁱ	-85.14 (15)	O1 ⁱⁱⁱ —Na2—C2—C4	-149.8 (3)
O1 ⁱⁱⁱ —Na4—O3—Na2 ⁱⁱⁱ	19.36 (13)	O1—Na2—C2—C4	-50.9 (3)
O15—Na4—O3—Na2 ⁱⁱⁱ	-179.53 (17)	O13—Na2—C2—C4	116.1 (3)
O2 ⁱⁱⁱ —Na4—O3—Na2 ⁱⁱⁱ	0.1 (2)	O13 ^{iv} —Na2—C2—C4	24.0 (3)
O4—Na4—O3—Na2 ⁱⁱⁱ	105.12 (15)	O3 ⁱⁱⁱ —Na2—C2—C4	-89.4 (3)
O9 ^{vii} —Na4—O3—Na2 ⁱⁱⁱ	121.3 (2)	O4—Na2—C2—C4	105.5 (4)
C1 ⁱⁱⁱ —Na4—O3—Na2 ⁱⁱⁱ	9.97 (16)	O1 ⁱⁱⁱ —Na2—C2—Na4	32.96 (12)
C2—Na4—O3—Na2 ⁱⁱⁱ	110.3 (3)	O1—Na2—C2—Na4	131.83 (17)
O1 ⁱⁱⁱ —Na2—O4—C2	-73.5 (3)	O13—Na2—C2—Na4	-61.18 (16)
O1—Na2—O4—C2	19.9 (3)	O13 ^{iv} —Na2—C2—Na4	-153.27 (14)
O13—Na2—O4—C2	-170.2 (3)	O3 ⁱⁱⁱ —Na2—C2—Na4	93.33 (19)
O13 ^{iv} —Na2—O4—C2	107.6 (3)	O4—Na2—C2—Na4	-71.7 (3)
O3 ⁱⁱⁱ —Na2—O4—C2	-28.0 (5)	O2—C1—C3—C4	178.1 (4)
O1 ⁱⁱⁱ —Na2—O4—Na3 ^{viii}	148.55 (13)	O1—C1—C3—C4	-3.1 (7)
O1—Na2—O4—Na3 ^{viii}	-118.03 (14)	O2—C1—C3—C8	-4.0 (7)
O13—Na2—O4—Na3 ^{viii}	51.86 (14)	O1—C1—C3—C8	174.9 (4)
O13 ^{iv} —Na2—O4—Na3 ^{viii}	-30.31 (16)	C8—C3—C4—C5	-1.1 (7)
O3 ⁱⁱⁱ —Na2—O4—Na3 ^{viii}	-166.0 (3)	C1—C3—C4—C5	176.8 (4)
C2—Na2—O4—Na3 ^{viii}	-138.0 (3)	C8—C3—C4—C2	-179.1 (4)
O1 ⁱⁱⁱ —Na2—O4—Na4	15.07 (13)	C1—C3—C4—C2	-1.1 (7)
O1—Na2—O4—Na4	108.50 (13)	O3—C2—C4—C5	97.5 (5)
O13—Na2—O4—Na4	-81.61 (13)	O4—C2—C4—C5	-84.2 (6)
O13 ^{iv} —Na2—O4—Na4	-163.78 (13)	O3—C2—C4—C3	-84.6 (6)
O3 ⁱⁱⁱ —Na2—O4—Na4	60.5 (4)	O4—C2—C4—C3	93.7 (5)
C2—Na2—O4—Na4	88.6 (3)	C3—C4—C5—C6	1.1 (8)
O10 ^{vi} —Na4—O4—C2	-13.9 (4)	C2—C4—C5—C6	179.1 (5)
O1 ⁱⁱⁱ —Na4—O4—C2	92.1 (3)	C3—C4—C5—N1	-178.9 (5)
O15—Na4—O4—C2	-99.0 (3)	C2—C4—C5—N1	-0.8 (7)
O3—Na4—O4—C2	5.2 (2)	O5—N1—C5—C4	-16.7 (9)
O2 ⁱⁱⁱ —Na4—O4—C2	124.1 (3)	O6—N1—C5—C4	-171.9 (10)
O9 ^{vii} —Na4—O4—C2	-163.6 (3)	O6'—N1—C5—C4	131.8 (11)
C1 ⁱⁱⁱ —Na4—O4—C2	105.6 (3)	O5—N1—C5—C6	163.4 (6)
O10 ^{vi} —Na4—O4—Na3 ^{viii}	146.4 (3)	O6—N1—C5—C6	8.1 (12)
O1 ⁱⁱⁱ —Na4—O4—Na3 ^{viii}	-107.6 (2)	O6'—N1—C5—C6	-48.2 (12)
O15—Na4—O4—Na3 ^{viii}	61.3 (2)	C4—C5—C6—C7	-0.2 (9)
O3—Na4—O4—Na3 ^{viii}	165.4 (3)	N1—C5—C6—C7	179.8 (5)
O2 ⁱⁱⁱ —Na4—O4—Na3 ^{viii}	-75.6 (3)	C5—C6—C7—C8	-0.7 (8)
O9 ^{vii} —Na4—O4—Na3 ^{viii}	-3.3 (2)	C6—C7—C8—C3	0.7 (8)
C1 ⁱⁱⁱ —Na4—O4—Na3 ^{viii}	-94.1 (2)	C4—C3—C8—C7	0.3 (7)

C2—Na4—O4—Na3 ^{viii}	160.3 (4)	C1—C3—C8—C7	-177.8 (5)
O10 ^{vi} —Na4—O4—Na2	-120.4 (3)	Na1 ⁱ —O8—C9—O7	100.8 (6)
O1 ⁱⁱⁱ —Na4—O4—Na2	-14.41 (13)	Na1—O8—C9—O7	-101.8 (6)
O15—Na4—O4—Na2	154.48 (18)	Na1 ⁱ —O8—C9—C11	-80.0 (7)
O3—Na4—O4—Na2	-101.40 (15)	Na1—O8—C9—C11	77.5 (6)
O2 ⁱⁱⁱ —Na4—O4—Na2	17.56 (19)	Na3—O7—C9—O8	177.4 (14)
O9 ^{vii} —Na4—O4—Na2	89.89 (13)	Na3—O7—C9—C11	-2(2)
O3—Na3—O7—C9	-102.8 (17)	Na4 ^x —O10—C10—O9	117.5 (8)
O4 ^v —Na3—O7—C9	115.6 (17)	Na1—O10—C10—O9	-80.8 (7)
O14—Na3—O7—C9	8.8 (18)	Na4 ^x —O10—C10—C12	-63.6 (10)
O11 ^{vi} —Na3—O7—C9	-16 (3)	Na1—O10—C10—C12	98.1 (5)
O2—Na1—O8—C9	32.0 (5)	Na1 ⁱ —O9—C10—O10	69.8 (7)
O8 ⁱ —Na1—O8—C9	-165.3 (5)	Na4 ^{ix} —O9—C10—O10	-86.9 (14)
O2 ⁱⁱ —Na1—O8—C9	-50.5 (7)	Na1 ⁱ —O9—C10—C12	-109.1 (4)
O10—Na1—O8—C9	-79.7 (5)	Na4 ^{ix} —O9—C10—C12	94.2 (13)
O9 ⁱ —Na1—O8—C9	119.9 (5)	O8—C9—C11—C16	-177.7 (5)
O2—Na1—O8—Na1 ⁱ	-162.62 (15)	O7—C9—C11—C16	1.6 (7)
O8 ⁱ —Na1—O8—Na1 ⁱ	0.0	O8—C9—C11—C12	1.6 (7)
O2 ⁱⁱ —Na1—O8—Na1 ⁱ	114.8 (3)	O7—C9—C11—C12	-179.1 (5)
O10—Na1—O8—Na1 ⁱ	85.65 (16)	C16—C11—C12—C13	1.8 (8)
O9 ⁱ —Na1—O8—Na1 ⁱ	-74.71 (15)	C9—C11—C12—C13	-177.5 (5)
O2—Na1—O10—C10	-105.2 (5)	C16—C11—C12—C10	-177.7 (5)
O8 ⁱ —Na1—O10—C10	80.0 (5)	C9—C11—C12—C10	3.0 (8)
O2 ⁱⁱ —Na1—O10—C10	176.1 (5)	O10—C10—C12—C13	88.0 (7)
O8—Na1—O10—C10	-16.8 (4)	O9—C10—C12—C13	-92.9 (7)
O9 ⁱ —Na1—O10—C10	45.6 (7)	O10—C10—C12—C11	-92.5 (6)
O2—Na1—O10—Na4 ^x	65.52 (19)	O9—C10—C12—C11	86.5 (7)
O8 ⁱ —Na1—O10—Na4 ^x	-109.31 (18)	C11—C12—C13—C14	-0.7 (9)
O2 ⁱⁱ —Na1—O10—Na4 ^x	-13.19 (16)	C10—C12—C13—C14	178.8 (6)
O8—Na1—O10—Na4 ^x	154.0 (2)	C11—C12—C13—N2	179.9 (5)
O9 ⁱ —Na1—O10—Na4 ^x	-143.7 (4)	C10—C12—C13—N2	-0.7 (9)
O12—N2—O11—Na3 ^x	6.4 (14)	O12—N2—C13—C14	7.1 (9)
O1 ⁱⁱⁱ —Na2—O13—Na2 ^{iv}	162.01 (16)	O11—N2—C13—C14	-171.5 (6)
O1—Na2—O13—Na2 ^{iv}	-72.9 (5)	O12—N2—C13—C12	-173.4 (6)
O13 ^{iv} —Na2—O13—Na2 ^{iv}	0.0	O11—N2—C13—C12	8.0 (9)
O3 ⁱⁱⁱ —Na2—O13—Na2 ^{iv}	84.68 (16)	C12—C13—C14—C15	-1.2 (10)
O4—Na2—O13—Na2 ^{iv}	-109.64 (15)	N2—C13—C14—C15	178.3 (6)
Na1—O2—C1—O1	99.1 (5)	C13—C14—C15—C16	1.8 (10)
Na1 ⁱⁱ —O2—C1—O1	-101.6 (5)	C12—C11—C16—C15	-1.2 (9)
Na4 ⁱⁱⁱ —O2—C1—O1	-4.7 (5)	C9—C11—C16—C15	178.1 (5)
Na1—O2—C1—C3	-82.1 (5)	C14—C15—C16—C11	-0.6 (10)

supplementary materials

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O13—H13A \cdots O7 ⁱⁱⁱ	0.85	1.94	2.789 (6)	180
O13—H13A \cdots O8 ⁱⁱⁱ	0.85	2.51	3.049 (5)	123
O13—H13B \cdots O10 ^{vii}	0.85	2.13	2.980 (6)	180
O13—H13B \cdots O9 ^{vii}	0.85	2.63	3.186 (6)	125
O14—H14B \cdots O6 ^{xi}	0.85	1.93	2.782 (13)	179
O14—H14B \cdots O6 ^{xi}	0.85	1.97	2.722 (15)	147
O14—H14C \cdots O6 ^v	0.85	1.45	2.29 (2)	166
O14—H14C \cdots N1 ^v	0.85	2.27	3.078 (10)	160
O14—H14C \cdots O6 ^v	0.85	2.55	3.388 (18)	171
O15—H15B \cdots O5	0.85	2.13	2.951 (7)	162
O15—H15C \cdots O9 ^{vii}	0.85	2.29	2.782 (8)	117
O15—H15C \cdots O11 ^{vii}	0.85	2.48	3.302 (8)	163

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

Fig. 1

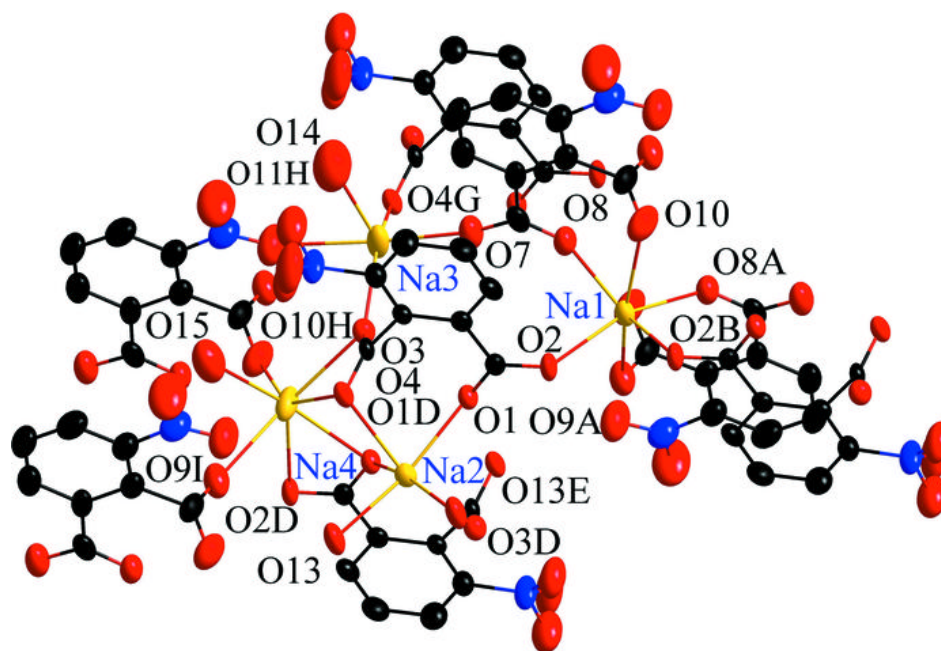


Fig. 2

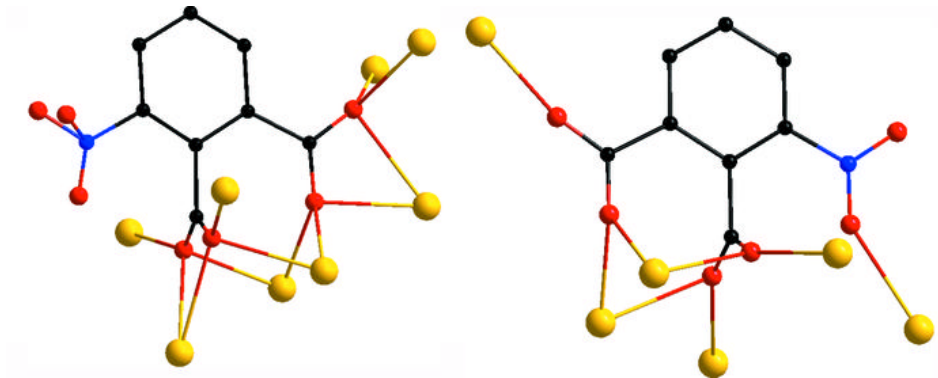


Fig. 3

