



Crystal structure of hexasodium tetraserinolium paratungstate B decahydrate, $[\text{Na}_6\{(\text{CH}_2\text{OH})_2\text{CHNH}_3\}_4][\text{W}_{12}\text{O}_{40}(\text{OH})_2]\cdot 10\text{H}_2\text{O}$

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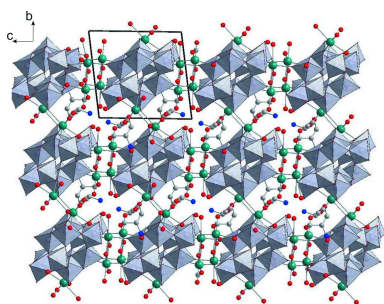
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The title polyoxometalate-based organic–inorganic hybrid compound, $[\text{Na}_6(\text{C}_3\text{H}_{10}\text{NO}_2)_4][\text{W}_{12}\text{O}_{40}(\text{OH})_2]\cdot 10\text{H}_2\text{O}$, consists of a di- μ_3 -hydroxido-tetra- μ_3 -oxido-octadeca- μ -oxido-octadecaoxidododecatungstate (paradodecatungstate B) anion, $[\text{W}_{12}\text{O}_{40}(\text{OH})_2]^{10-}$, and six sodium cations coordinated by the oxygen ions of the polyanions, serinol ligands protonated at the N atom, and water molecules. The centrosymmetric paratungstate B anion shows characteristic features in terms of bond lengths and angles. The three-dimensional framework structure is established by bonding of the sodium cations with oxygen ions of the paratungstate B anions and a network consisting of N–H \cdots O and O–H \cdots O hydrogen bonds of medium strength between the protonated serinol cations, water molecules and the paratungstate B anions. The title compound was also characterized by means of elemental analysis, IR spectroscopy and thermogravimetric analysis.

1. Chemical context

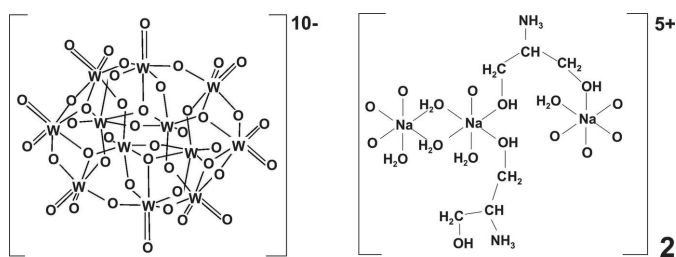
Polyoxometalates (POMs) are discrete anionic molecular clusters of metal oxide entities, which usually consist of transition metals of groups V and VI in their highest oxidation states. POMs exist at a unique interface between monomeric oxometalates and polymeric metal oxides and have a wide range of applications (Pope, 1983; Gumerova & Rompel, 2020). To date, a variety of strategies have been developed and used to build POM-based hybrid materials by varying the reaction conditions such as the type of addenda ions, organic ligands, pH, solvents, the molar ratio of the starting materials or the reaction environments. The $[\text{W}_{12}\text{O}_{40}(\text{OH})_2]^{10-}$ paratungstate B anion is stable in aqueous acidic solution and has a cluster-like structure of twelve W-centered distorted octahedra $\{\text{WO}_6\}$ (Evans & Rollins, 1976; Pope, 1983). Due to its high surface charge density $q/m = 0.83$ (q = net charge, m = number of metal ions), the paratungstate B anion can act as a multidentate ligand for alkaline (Peresyphkina *et al.*, 2014) or transition-metal cations (Radio *et al.*, 2010, 2011; Gumerova *et al.*, 2015, 2018) and as a precursor for the synthesis of catalytically active sandwich-type polyoxotungstates (POTs) (Sokolov *et al.*, 2012).

A search in the Cambridge Structural Database (version 5.42, update of November 2021; Groom *et al.*, 2016) revealed that seventeen organic–inorganic hybrid paratungstates B have been structurally characterized so far. We are expanding



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the class of hybrid paratungstates by using serinol ($C_3H_9NO_2$; 2-amino-1,3-propanediol), which has not previously been used in its protonated form as a counter-cation for paratungstates and can coordinate to metal cations in different ways *via* its $-NH_2$ or $HOCH_2-$ groups and thus influences both the structure and the properties of the compound significantly (Sifaki *et al.*, 2021). Serinol is a very stable, readily water-soluble, non-toxic, odorless, biodegradable compound that is used as a versatile starting material in organic synthesis and as an additive for material applications, such as composite materials (Barbera *et al.*, 2020; Andreessen & Steinbüchel, 2011). In POM synthesis, due to its amino group, serinol can be regarded as an alkoxylation ligand and/or as a buffer compound ($pK_a = 12.2$; Chemicalbook, 2021). With its protonated amine group it can also act as a counter-cation.



2. Structural commentary

The asymmetric unit of the title compound is made up of half of the paratungstate B polyoxoanion, $[W_{12}O_{40}(OH)_2]^{10-}$, three sodium cations coordinated by water molecules, terminal oxygen atoms of the paratungstate anion and the oxygen atoms of $HOCH_2-$ groups from two serinol cations protonated at the N atoms $[(CH_2OH)_2CHNH_3^+]$ (Fig. 1). An inversion center creates a full unit with formula $[Na_6((CH_2OH)_2CHNH_3)_4][W_{12}O_{40}(OH)_2] \cdot 10H_2O$ (Fig. 2). The centrosymmetric paratungstate B anion $[W_{12}O_{40}(OH)_2]^{10-}$ is structurally very similar to previously described ones (Radio *et al.*, 2010, 2011; Gumerova *et al.*, 2015, 2018) and consists of four groups, *viz.* two $\{W_3O_{13}\}$ and two $\{W_3O_{14}\}$ units, with common vertices (Fig. 1). In the $\{W_3O_{13}\}$ groups each $\{WO_6\}$ octahedron has a terminal oxygen atom, while in the $\{W_3O_{14}\}$ units each $\{WO_6\}$ octahedron has two unshared oxygen ions (Fig. 1). The oxygen atoms associated with the central W ions can be divided into three groups: 1) terminal oxygen ions (O_t), each bonded to a W ion (magenta labeling in Fig. 1); 2) bridging oxygen ions (O_{db}), each connected to two W ions (blue labeling in Fig. 1); 3) triply bridging oxygen ions (O_{tb}) linked to three W ions (green labeling in Fig. 1).

3. Supramolecular features

The paratungstate B anion is bound to twelve Na^+ cations *via* both terminal (O_t) and bridging oxygen atoms (O_{db}). Each of the surrounding four Na1 cations and six Na3 cations are coordinated by the O atoms of two polyanions, while two Na2

cations are bound to one terminal oxygen atom of the polyanion and to the O atoms of two serinoliium cations, which are further connected to the Na3 cations *via* the $HOCH_2-$ groups. Thus, a three-dimensional framework is established in the crystal structure by connecting paratungstate B anions through $O_t-Na1-O_t$, $O_t-Na3-O_t$ and O_t-Na2 -serinoliium- $Na3-O_t$ bridges (Fig. 2). An intimate network of $N-H \cdots O$ and $O-H \cdots O$ hydrogen bonds of medium strength between the protonated serinol ligands, polyoxoanions and water molecules consolidates the crystal packing (Table 1).

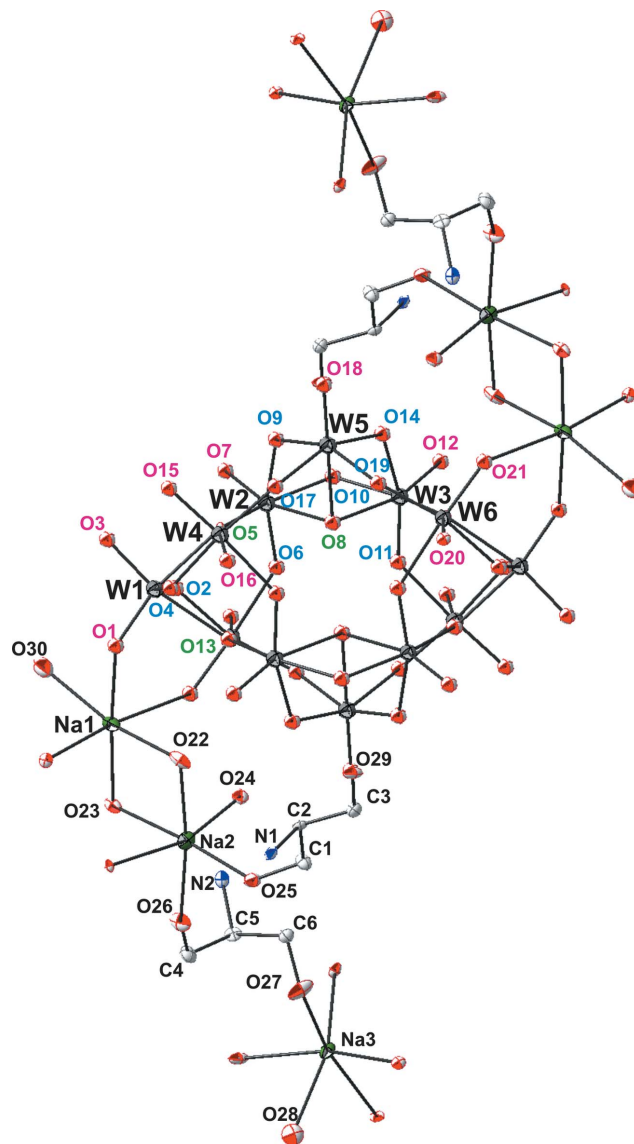


Figure 1
The principal building units in the crystal structure of $[Na_6((CH_2OH)_2CHNH_3)_4][W_{12}O_{40}(OH)_2] \cdot 10H_2O$. The asymmetric unit was doubled considering the inversion center, and coordination spheres for all cations were completed. Labeling of all atoms of asymmetric unit is shown; non-labeled atoms are generated by symmetry operation $-x + 1, -y + 1, -z + 1$. The oxygen atoms in the paratungstate B anion are labeled according to their coordination mode: magenta for the terminal, blue for the double bridging, green for the triply bridging oxygen ions. Displacement ellipsoids are drawn at the 50% probability level. Color code: W, dark gray; Na, green; C, gray; N, blue; O, red.

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O22—H22A \cdots O1 ⁱ	0.67 (7)	2.11 (7)	2.757 (5)	163 (8)
O23—H23A \cdots O16 ⁱ	0.78 (7)	2.04 (7)	2.802 (5)	165 (7)
O23—H23B \cdots O16 ⁱⁱ	0.91 (7)	1.91 (7)	2.811 (5)	175 (6)
O25—H25 \cdots O3 ⁱⁱⁱ	0.73 (6)	2.05 (7)	2.761 (5)	164 (7)
O26—H26 \cdots O15 ⁱ	0.88 (7)	1.88 (7)	2.730 (5)	160 (6)
O27—H27 \cdots O2 ⁱⁱⁱ	0.67 (7)	2.04 (7)	2.696 (5)	165 (8)
O28—H28A \cdots O21 ^{iv}	0.75 (8)	2.01 (8)	2.735 (6)	160 (8)
O29—H29 \cdots O10 ^v	0.81 (6)	1.91 (7)	2.692 (5)	163 (6)
N1—H1B \cdots O15 ⁱⁱⁱ	0.91	1.82	2.723 (5)	170
N2—H2B \cdots O24	0.91	1.96	2.867 (6)	172

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

4. Database survey

A search in the Cambridge Structural Database (CSD; version 5.42, update of November 2021; Groom *et al.*, 2016) indicated 17 structures with the formula search [$W_{12}O_{42}$] or [$W_{12}O_{40}(OH)_2$], including eleven structures with only organic cations and six structures with organic and transition-metal cations. The 17 compounds deposited in the CSD all crystallize in centrosymmetric space groups, and the bond lengths in the paratungstate B anion are very similar to those observed in the title structure. A similar structure containing sodium and a protonated imidazole as counter-cations, *viz.* $Na_2(HIm)_8[Na_6((CH_2OH)_2CHNH_3)_4[W_{12}O_{40}(OH)_2] \cdot 10H_2O$ (HIm: imidazolium), is comprised of infinite inorganic chains built up from [$W_{12}O_{40}(OH)_2$]¹⁰⁻ anions and sodium cations. Adjacent chains are further connected by hydrogen-bonding interactions between imidazolium cations, water molecules, and polyoxoanions (Chaalía *et al.*, 2012).

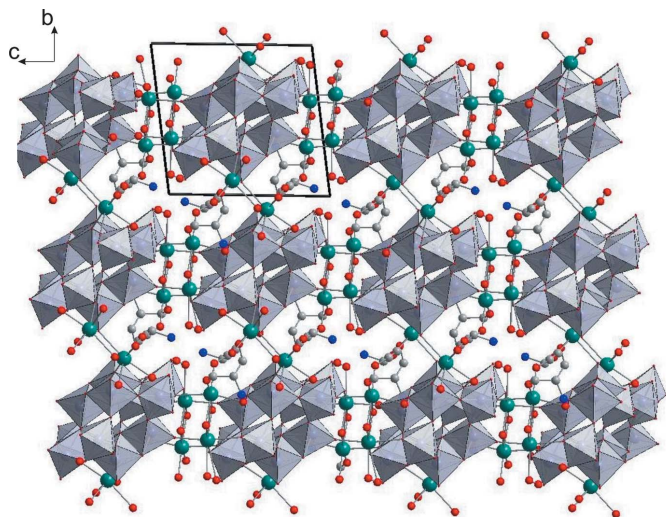


Figure 2
The crystal packing of $[Na_6((CH_2OH)_2CHNH_3)_4][W_{12}O_{40}(OH)_2] \cdot 10H_2O$ viewed along the a axis. Color code: $\{WO_6\}$: gray octahedra; Na: green; C: gray; N: blue; O: red.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Na_6((C_{12}H_{40}N_4O_8)]-[W_{12}O_{40}(OH)_2] \cdot 10H_2O$
M_r	3566.79
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	200
a, b, c (Å)	12.0541 (8), 12.0821 (8), 12.7050 (8)
α, β, γ (°)	73.180 (2), 65.308 (2), 64.345 (2)
V (Å ³)	1502.07 (17)
Z	1
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	23.04
Crystal size (mm)	0.05 × 0.05 × 0.05
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (SADABS; Krause <i>et al.</i> , 2015)
T_{min}, T_{max}	0.245, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	39702, 5490, 5404
R_{int}	0.038
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.017, 0.043, 1.25
No. of reflections	5490
No. of parameters	472
No. of restraints	6
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.99, -1.62

Computer programs: APEX3 and SAINT (Bruker, 2016), olex2.solve (Bourhis *et al.*, 2015), SHELXL (Sheldrick, 2015) and OLEX2 (Dolomanov *et al.*, 2009).

5. Synthesis and crystallization

To obtain the title compound, $Na_2WO_4 \cdot 2H_2O$ (0.495 g, 1.5 mmol) was dissolved in 5 ml of distilled water and acidified with 1 M HCl to pH 3. Serinol (0.075 g, 0.8 mmol) was then added to the acidified orthotungstate solution, which increased the pH to 6.7. The reaction mixture was then heated to 363 K and stirred for 1 h, cooled to room temperature and left covered with parafilm. Colorless block-shaped crystals were filtered off after one week from the mother liquor, washed with water and ethanol and then air-dried (yield 0.12 g; 27%, based on W). Elemental analysis (%) for $C_{12}H_{62}N_4Na_6O_{60}W_{12}$ (calculated): C 3.23 (4.04), H 1.71 (1.75), N 1.41 (1.57), O 26.68 (26.92). FT-IR (cm⁻¹): 3340 (*s*), 2952 (*sh*), 2889 (*sh*), 1614 (*s*), 1450 (*m*), 1066 (*w*), 1037 (*m*), 996 (*w*), 930 (*s*), 896 (*s*), 794 (*s*), 681 (*s*), 620 (*m*), 478 (*s*), 456 (*s*), 428 (*s*), 310 (*s*). Mass loss observed in thermogravimetric analysis in the temperature range 298–1073 K (calculated for four protonated serinol ligands, ten crystal water molecules and one water molecule from the anion): 16.67% (16.78%).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The positions of the H atoms of the

water molecules (O22, O23, O24, O28) were obtained by difference-Fourier techniques and were refined with free isotropic displacement parameters and O–H distances restrained to 0.95 (2) Å. The disordered water molecule (O30) was refined with two positions (O30A and O30B), with free occupancy factors to a total of 100%. H atoms bound to N or C atoms were placed in idealized positions (N–H = 0.91 Å and C–H = 0.99 or 1.00 Å for CH₂ and CH groups, respectively) and refined in riding modes, with $U_{\text{iso}}(\text{H})$ values set to $1.5U_{\text{eq}}(\text{N})$ or to $1.2U_{\text{eq}}(\text{C})$. Three H-atom positions could not be included in the final model: two H-atom positions from the disordered water molecule (O30A and O30B), and one H atom that should be located inside the paratungstate B anion on the triply bridging O8 atom, which was previously proven by neutron diffraction (Evans & Prince, 1983).

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supporting information

Acta Cryst. (2022). E78, 207-210 [https://doi.org/10.1107/S2056989022000457]

Crystal structure of hexasodium tetraserinolium paratungstate B decahydrate, [Na₆{(CH₂OH)₂CHNH₃}₄][W₁₂O₄₀(OH)₂]·10H₂O

Kleanthi Sifaki, Nadiia I. Gumerova, Gerald Giester and Annette Rompel

Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINTE* (Bruker, 2016); data reduction: *SAINTE* (Bruker, 2016); program(s) used to solve structure: *olex2.solve* (Bourhis *et al.*, 2015); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Hexasodium tetraserinolium di- μ_3 -hydroxido-tetra- μ_3 -oxido-octadeca- μ -oxido-octadecaoxidododecatungstate decahydrate

Crystal data

[Na₆(C₃H₁₀NO₂)₄][W₁₂O₄₀(OH)₂]·10H₂O

$M_r = 3566.79$

Triclinic, $P\bar{1}$

$a = 12.0541$ (8) Å

$b = 12.0821$ (8) Å

$c = 12.7050$ (8) Å

$\alpha = 73.180$ (2)°

$\beta = 65.308$ (2)°

$\gamma = 64.345$ (2)°

$V = 1502.07$ (17) Å³

$Z = 1$

$F(000) = 1596$

$D_x = 3.943$ Mg m⁻³

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

Cell parameters from 9742 reflections

$\theta = 2.2$ – 33.1 °

$\mu = 23.04$ mm⁻¹

$T = 200$ K

Block, clear colourless

$0.05 \times 0.05 \times 0.05$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: sealed x-ray tube

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Krause *et al.*, 2015)

$T_{\min} = 0.245$, $T_{\max} = 0.747$

39702 measured reflections

5490 independent reflections

5404 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 25.4$ °, $\theta_{\min} = 2.2$ °

$h = -14 \rightarrow 14$

$k = -14 \rightarrow 14$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

$wR(F^2) = 0.043$

$S = 1.25$

5490 reflections

472 parameters

6 restraints

Primary atom site location: iterative

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0095P)^2 + 5.2022P]$

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

$$(\Delta/\sigma)_{\max} = 0.002$$

$$\Delta\rho_{\max} = 0.99 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.61 \text{ e } \text{\AA}^{-3}$$

Special details

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

Refinement. olex2_refinement_description 1. Fixed Uiso At 1.2 times of: All C(H) groups, All C(H,H) groups, All O(H) groups At 1.5 times of: All N(H,H,H) groups, All O(H,H) groups 2. Uiso/Uanis restraints and constraints O30A ~ O30B: within 2A with sigma of 0.04 and sigma for terminal atoms of 0.08 within 2A 3. Others Sof(O30A)=1-FVAR(1) Sof(O30B)=FVAR(1) 4.a Ternary CH refined with riding coordinates: C2(H2), C5(H5) 4.b Secondary CH2 refined with riding coordinates: C1(H1D,H1E), C3(H3A,H3B), C4(H4A,H4B), C6(H6A,H6B) 4.c Idealised Me refined as rotating group: N1(H1A,H1B,H1C), N2(H2A,H2B,H2C)

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
W1	0.42560 (2)	0.74680 (2)	0.16437 (2)	0.00609 (5)	
W2	0.38107 (2)	0.76530 (2)	0.46722 (2)	0.00492 (5)	
W3	0.43557 (2)	0.58129 (2)	0.71174 (2)	0.00468 (5)	
W4	0.70356 (2)	0.65943 (2)	0.21152 (2)	0.00538 (5)	
W5	0.66091 (2)	0.68816 (2)	0.50890 (2)	0.00550 (5)	
W6	0.79490 (2)	0.36191 (2)	0.62441 (2)	0.00589 (5)	
Na1	0.2506 (2)	0.6297 (2)	0.05508 (18)	0.0188 (4)	
Na2	0.2644 (2)	0.32264 (19)	0.11025 (19)	0.0195 (5)	
Na3	-0.1267 (2)	-0.09506 (18)	0.40382 (18)	0.0166 (4)	
O1	0.3975 (3)	0.7093 (3)	0.0574 (3)	0.0103 (7)	
O2	0.2557 (3)	0.7800 (3)	0.2802 (3)	0.0091 (7)	
O3	0.4137 (3)	0.9003 (3)	0.1124 (3)	0.0119 (7)	
O4	0.6114 (3)	0.6631 (3)	0.1144 (3)	0.0077 (7)	
O5	0.4911 (3)	0.7439 (3)	0.3094 (3)	0.0068 (6)	
O6	0.2927 (3)	0.6703 (3)	0.4788 (3)	0.0098 (7)	
O7	0.2695 (3)	0.9141 (3)	0.4594 (3)	0.0114 (7)	
O8	0.5351 (3)	0.5906 (3)	0.5163 (3)	0.0061 (6)	
O9	0.5209 (3)	0.8146 (3)	0.4727 (3)	0.0070 (6)	
O10	0.3393 (3)	0.7379 (3)	0.6367 (3)	0.0058 (6)	
O11	0.3480 (3)	0.5000 (3)	0.6978 (3)	0.0096 (7)	
O12	0.3556 (3)	0.6163 (3)	0.8530 (3)	0.0108 (7)	
O13	0.5815 (3)	0.4361 (3)	0.7240 (3)	0.0077 (7)	
O14	0.5651 (3)	0.6687 (3)	0.6669 (3)	0.0081 (7)	
O15	0.7266 (3)	0.7992 (3)	0.1354 (3)	0.0116 (7)	
O16	0.8550 (3)	0.5513 (3)	0.1411 (3)	0.0092 (7)	
O17	0.7336 (3)	0.6599 (3)	0.3434 (3)	0.0088 (7)	
O18	0.7542 (3)	0.7663 (3)	0.5051 (3)	0.0113 (7)	
O19	0.7760 (3)	0.5146 (3)	0.5302 (3)	0.0090 (7)	
O20	0.9485 (3)	0.2716 (3)	0.5427 (3)	0.0104 (7)	
O21	0.8303 (3)	0.3935 (3)	0.7318 (3)	0.0113 (7)	
O22	0.3854 (4)	0.4292 (4)	0.1050 (4)	0.0193 (9)	

H22A	0.447 (7)	0.401 (7)	0.068 (6)	0.029*	
H22B	0.397 (7)	0.418 (6)	0.174 (6)	0.029*	
O23	0.1180 (4)	0.5313 (4)	0.0558 (3)	0.0156 (8)	
H23A	0.138 (7)	0.511 (6)	-0.005 (6)	0.023*	
H23B	0.032 (7)	0.540 (6)	0.087 (6)	0.023*	
O24	0.1349 (4)	0.3728 (4)	0.3060 (4)	0.0203 (9)	
H24A	0.155 (7)	0.334 (6)	0.368 (6)	0.030*	
H24B	0.143 (8)	0.423 (7)	0.300 (7)	0.030*	
O25	0.3957 (4)	0.1204 (3)	0.1540 (4)	0.0187 (8)	
H25	0.390 (7)	0.070 (6)	0.137 (6)	0.022*	
O26	0.1374 (4)	0.2320 (4)	0.0943 (4)	0.0260 (9)	
H26	0.161 (7)	0.230 (6)	0.018 (6)	0.031*	
O27	0.0421 (4)	-0.0116 (4)	0.3221 (4)	0.0269 (10)	
H27	0.101 (7)	-0.057 (7)	0.313 (7)	0.032*	
O28	-0.0593 (4)	-0.2184 (4)	0.2434 (4)	0.0249 (9)	
H28A	-0.002 (8)	-0.277 (7)	0.247 (7)	0.037*	
H28B	-0.118 (8)	-0.237 (7)	0.254 (7)	0.037*	
O29	0.7568 (4)	0.0498 (4)	0.2733 (4)	0.0182 (8)	
H29	0.735 (6)	0.119 (6)	0.287 (6)	0.022*	
N1	0.6651 (4)	0.0491 (4)	0.0932 (4)	0.0135 (9)	
H1A	0.620503	0.082305	0.041998	0.020*	
H1B	0.694572	-0.035013	0.100535	0.020*	
H1C	0.734329	0.074419	0.066187	0.020*	
N2	-0.0763 (4)	0.3259 (4)	0.3009 (4)	0.0123 (9)	
H2A	-0.109556	0.387738	0.249053	0.018*	
H2B	-0.004148	0.332753	0.301037	0.018*	
H2C	-0.137617	0.331930	0.373678	0.018*	
C1	0.4520 (5)	0.0653 (5)	0.2438 (5)	0.0165 (11)	
H1D	0.472355	-0.025201	0.257484	0.020*	
H1E	0.388654	0.099437	0.317357	0.020*	
C2	0.5768 (5)	0.0910 (4)	0.2092 (4)	0.0107 (10)	
H2	0.554428	0.182003	0.203335	0.013*	
C3	0.6425 (5)	0.0233 (5)	0.2991 (5)	0.0162 (11)	
H3A	0.580025	0.047163	0.376761	0.019*	
H3B	0.667484	-0.066948	0.302642	0.019*	
C4	0.0445 (5)	0.1009 (4)	0.3328 (5)	0.0147 (11)	
H4A	0.135127	0.100118	0.299111	0.018*	
H4B	0.008737	0.113215	0.415968	0.018*	
C5	-0.0397 (5)	0.2030 (4)	0.2660 (4)	0.0127 (10)	
H5	-0.122474	0.187396	0.290152	0.015*	
C6	0.0209 (5)	0.2060 (5)	0.1362 (5)	0.0187 (11)	
H6A	0.041210	0.124953	0.115096	0.022*	
H6B	-0.041824	0.270265	0.098737	0.022*	
O30B	0.9054 (14)	0.105 (2)	-0.080 (3)	0.048 (6)	0.48 (5)
O30A	0.8950 (11)	0.1308 (12)	-0.026 (2)	0.035 (5)	0.52 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
W1	0.00509 (9)	0.00748 (9)	0.00526 (10)	-0.00281 (7)	-0.00147 (7)	0.00021 (7)
W2	0.00343 (9)	0.00618 (9)	0.00481 (10)	-0.00225 (7)	-0.00065 (7)	-0.00056 (7)
W3	0.00345 (9)	0.00601 (9)	0.00437 (10)	-0.00258 (7)	-0.00026 (7)	-0.00063 (7)
W4	0.00401 (9)	0.00709 (9)	0.00488 (10)	-0.00339 (7)	0.00000 (7)	-0.00063 (7)
W5	0.00430 (9)	0.00760 (9)	0.00575 (10)	-0.00427 (7)	-0.00078 (7)	-0.00051 (7)
W6	0.00366 (9)	0.00720 (9)	0.00671 (10)	-0.00237 (7)	-0.00130 (7)	-0.00083 (7)
Na1	0.0206 (11)	0.0287 (12)	0.0124 (10)	-0.0171 (10)	-0.0008 (9)	-0.0044 (9)
Na2	0.0148 (10)	0.0173 (10)	0.0248 (12)	-0.0047 (9)	-0.0068 (9)	-0.0020 (9)
Na3	0.0135 (10)	0.0150 (10)	0.0215 (11)	-0.0074 (8)	-0.0073 (9)	0.0029 (8)
O1	0.0080 (16)	0.0132 (17)	0.0082 (17)	-0.0048 (14)	0.0000 (13)	-0.0018 (13)
O2	0.0094 (16)	0.0104 (16)	0.0090 (17)	-0.0048 (14)	-0.0031 (14)	-0.0011 (13)
O3	0.0143 (18)	0.0096 (16)	0.0126 (18)	-0.0055 (14)	-0.0058 (15)	0.0012 (13)
O4	0.0070 (16)	0.0101 (16)	0.0045 (16)	-0.0027 (13)	-0.0010 (13)	-0.0010 (13)
O5	0.0052 (15)	0.0071 (15)	0.0071 (16)	-0.0013 (13)	-0.0026 (13)	-0.0001 (12)
O6	0.0111 (17)	0.0153 (17)	0.0043 (16)	-0.0079 (14)	-0.0004 (13)	-0.0017 (13)
O7	0.0098 (17)	0.0085 (16)	0.0120 (18)	-0.0016 (14)	-0.0022 (14)	-0.0008 (13)
O8	0.0032 (15)	0.0079 (15)	0.0069 (16)	-0.0031 (13)	-0.0001 (13)	-0.0012 (12)
O9	0.0036 (15)	0.0071 (15)	0.0088 (16)	-0.0021 (13)	-0.0003 (13)	-0.0013 (12)
O10	0.0017 (15)	0.0078 (15)	0.0059 (16)	-0.0012 (12)	0.0006 (12)	-0.0019 (12)
O11	0.0103 (17)	0.0112 (16)	0.0078 (17)	-0.0075 (14)	-0.0010 (14)	0.0006 (13)
O12	0.0072 (16)	0.0120 (16)	0.0103 (18)	-0.0029 (14)	-0.0008 (14)	-0.0017 (13)
O13	0.0056 (16)	0.0111 (16)	0.0066 (16)	-0.0041 (13)	-0.0014 (13)	-0.0009 (13)
O14	0.0071 (16)	0.0094 (16)	0.0085 (17)	-0.0042 (13)	-0.0022 (13)	-0.0011 (13)
O15	0.0125 (17)	0.0122 (17)	0.0109 (17)	-0.0074 (14)	-0.0018 (14)	-0.0011 (13)
O16	0.0040 (15)	0.0124 (16)	0.0095 (17)	-0.0036 (13)	0.0010 (13)	-0.0029 (13)
O17	0.0076 (16)	0.0137 (16)	0.0076 (17)	-0.0069 (14)	-0.0009 (13)	-0.0027 (13)
O18	0.0126 (17)	0.0139 (17)	0.0102 (17)	-0.0105 (15)	-0.0005 (14)	-0.0012 (14)
O19	0.0063 (16)	0.0088 (16)	0.0090 (17)	-0.0021 (13)	-0.0022 (13)	0.0011 (13)
O20	0.0071 (16)	0.0113 (16)	0.0126 (18)	-0.0037 (14)	-0.0027 (14)	-0.0018 (13)
O21	0.0102 (17)	0.0154 (17)	0.0099 (17)	-0.0070 (14)	-0.0016 (14)	-0.0026 (14)
O22	0.0098 (19)	0.033 (2)	0.015 (2)	-0.0058 (18)	0.0002 (16)	-0.0137 (18)
O23	0.0132 (19)	0.026 (2)	0.0117 (19)	-0.0114 (17)	-0.0014 (16)	-0.0046 (16)
O24	0.027 (2)	0.024 (2)	0.019 (2)	-0.0165 (19)	-0.0117 (18)	0.0022 (18)
O25	0.019 (2)	0.0128 (18)	0.028 (2)	-0.0040 (16)	-0.0139 (17)	-0.0027 (16)
O26	0.031 (2)	0.039 (2)	0.013 (2)	-0.024 (2)	0.0030 (18)	-0.0063 (18)
O27	0.013 (2)	0.012 (2)	0.052 (3)	-0.0004 (16)	-0.010 (2)	-0.0046 (19)
O28	0.013 (2)	0.028 (2)	0.033 (2)	-0.0048 (18)	-0.0076 (19)	-0.0059 (19)
O29	0.0168 (19)	0.0132 (18)	0.032 (2)	-0.0031 (16)	-0.0137 (17)	-0.0076 (16)
N1	0.018 (2)	0.010 (2)	0.010 (2)	-0.0072 (18)	-0.0023 (18)	0.0010 (16)
N2	0.008 (2)	0.015 (2)	0.011 (2)	-0.0023 (17)	-0.0015 (17)	-0.0014 (17)
C1	0.014 (3)	0.022 (3)	0.015 (3)	-0.008 (2)	-0.005 (2)	-0.002 (2)
C2	0.010 (2)	0.007 (2)	0.013 (3)	-0.0018 (19)	-0.003 (2)	-0.0036 (19)
C3	0.019 (3)	0.013 (2)	0.017 (3)	-0.005 (2)	-0.008 (2)	-0.001 (2)
C4	0.014 (3)	0.013 (2)	0.016 (3)	-0.003 (2)	-0.005 (2)	-0.003 (2)
C5	0.005 (2)	0.016 (2)	0.018 (3)	-0.006 (2)	-0.002 (2)	-0.003 (2)

C6	0.016 (3)	0.021 (3)	0.022 (3)	-0.004 (2)	-0.012 (2)	-0.003 (2)
O30B	0.048 (7)	0.092 (10)	0.028 (11)	-0.052 (7)	-0.011 (7)	0.000 (9)
O30A	0.046 (6)	0.048 (7)	0.027 (10)	-0.030 (5)	-0.017 (5)	0.001 (5)

Geometric parameters (Å, °)

W1—O1	1.743 (3)	Na2—O25	2.321 (4)
W1—O2	1.906 (3)	Na2—O26	2.328 (5)
W1—O3	1.742 (3)	Na3—O7 ^{iv}	2.684 (4)
W1—O4	1.914 (3)	Na3—O18 ^v	2.411 (4)
W1—O5	2.274 (3)	Na3—O20 ^{vi}	2.458 (4)
W1—O13 ⁱ	2.270 (3)	Na3—O27	2.375 (5)
W2—O5	1.905 (3)	Na3—O28	2.522 (5)
W2—O6	1.818 (3)	Na3—O29 ^{vii}	2.434 (4)
W2—O7	1.723 (3)	O22—H22A	0.68 (7)
W2—O8	2.259 (3)	O22—H22B	0.90 (7)
W2—O9	2.047 (3)	O23—H23A	0.78 (7)
W2—O10	1.956 (3)	O23—H23B	0.91 (7)
W3—O8	2.255 (3)	O24—H24A	0.86 (7)
W3—O10	1.953 (3)	O24—H24B	0.63 (7)
W3—O11	1.807 (3)	O25—H25	0.73 (7)
W3—O12	1.723 (3)	O25—C1	1.422 (7)
W3—O13	1.896 (3)	O26—H26	0.89 (7)
W3—O14	2.052 (3)	O26—C6	1.420 (7)
W4—O4	1.957 (3)	O27—H27	0.67 (7)
W4—O5	2.227 (3)	O27—C4	1.418 (6)
W4—O11 ⁱ	2.140 (3)	O28—H28A	0.75 (8)
W4—O15	1.761 (3)	O28—H28B	0.79 (8)
W4—O16	1.757 (3)	O29—H29	0.81 (7)
W4—O17	1.858 (3)	O29—C3	1.432 (6)
W5—O8	2.251 (3)	N1—H1A	0.9100
W5—O9	1.845 (3)	N1—H1B	0.9100
W5—O14	1.856 (3)	N1—H1C	0.9100
W5—O17	1.980 (3)	N1—C2	1.489 (6)
W5—O18	1.734 (3)	N2—H2A	0.9100
W5—O19	1.965 (3)	N2—H2B	0.9100
W6—O2 ⁱ	1.966 (3)	N2—H2C	0.9100
W6—O6 ⁱ	2.189 (3)	N2—C5	1.498 (6)
W6—O13	2.228 (3)	C1—H1D	0.9900
W6—O19	1.864 (3)	C1—H1E	0.9900
W6—O20	1.731 (3)	C1—C2	1.525 (7)
W6—O21	1.765 (3)	C2—H2	1.0000
Na1—Na2	3.513 (3)	C2—C3	1.502 (7)
Na1—O1	2.365 (4)	C3—H3A	0.9900
Na1—O12 ⁱⁱ	2.360 (4)	C3—H3B	0.9900
Na1—O21 ⁱ	2.440 (4)	C4—H4A	0.9900
Na1—O22	2.339 (5)	C4—H4B	0.9900
Na1—O23	2.365 (4)	C4—C5	1.517 (7)

Na1—O30B ⁱⁱⁱ	2.96 (3)	C5—H5	1.0000
Na1—O30A ⁱⁱⁱ	2.668 (12)	C5—C6	1.496 (7)
Na2—O4 ⁱⁱⁱ	2.606 (4)	C6—H6A	0.9900
Na2—O22	2.299 (4)	C6—H6B	0.9900
Na2—O23	2.471 (4)	O30B—O30A	0.772 (14)
Na2—O24	2.409 (5)		
O1—W1—O2	99.34 (14)	O24—Na2—Na1	82.89 (12)
O1—W1—O4	97.41 (14)	O24—Na2—O4 ⁱⁱⁱ	163.49 (15)
O1—W1—O5	165.62 (13)	O24—Na2—H22A	99.6 (17)
O1—W1—O13 ⁱ	90.34 (13)	O24—Na2—O23	83.19 (15)
O2—W1—O4	152.13 (14)	O24—Na2—H23A	99.9 (16)
O2—W1—O5	85.31 (13)	O24—Na2—H24B	14.0 (16)
O2—W1—O13 ⁱ	72.81 (12)	O25—Na2—Na1	141.81 (13)
O3—W1—O1	101.50 (15)	O25—Na2—O4 ⁱⁱⁱ	95.66 (14)
O3—W1—O2	97.16 (15)	O25—Na2—H22A	91.2 (16)
O3—W1—O4	101.13 (15)	O25—Na2—O23	175.41 (17)
O3—W1—O5	91.33 (14)	O25—Na2—H23A	158.6 (16)
O3—W1—O13 ⁱ	165.73 (14)	O25—Na2—O24	99.07 (17)
O4—W1—O5	73.49 (12)	O25—Na2—H24B	107.6 (17)
O4—W1—O13 ⁱ	85.00 (12)	O25—Na2—O26	84.84 (16)
O13 ⁱ —W1—O5	77.96 (11)	O26—Na2—Na1	133.01 (14)
O5—W2—O8	85.93 (12)	O26—Na2—O4 ⁱⁱⁱ	91.27 (14)
O5—W2—O9	84.55 (13)	O26—Na2—H22A	162.9 (17)
O5—W2—O10	155.42 (13)	O26—Na2—O23	90.92 (16)
O6—W2—O5	95.34 (14)	O26—Na2—H23A	82.9 (15)
O6—W2—O8	88.22 (13)	O26—Na2—O24	97.41 (16)
O6—W2—O9	160.42 (14)	O26—Na2—H24B	108.9 (17)
O6—W2—O10	91.33 (13)	O18 ^v —Na3—O7 ^{iv}	99.45 (13)
O7—W2—O5	104.66 (15)	O18 ^v —Na3—O20 ^{vi}	82.68 (13)
O7—W2—O6	103.57 (16)	O18 ^v —Na3—O28	79.23 (14)
O7—W2—O8	163.09 (14)	O18 ^v —Na3—O29 ^{vii}	104.03 (14)
O7—W2—O9	95.34 (14)	O20 ^{vi} —Na3—O7 ^{iv}	118.30 (13)
O7—W2—O10	96.66 (14)	O20 ^{vi} —Na3—O28	82.88 (14)
O9—W2—O8	72.23 (11)	O27—Na3—O7 ^{iv}	88.69 (14)
O10—W2—O8	70.63 (12)	O27—Na3—O18 ^v	162.22 (16)
O10—W2—O9	81.39 (13)	O27—Na3—O20 ^{vi}	79.54 (14)
O10—W3—O8	70.79 (12)	O27—Na3—O28	98.85 (17)
O10—W3—O14	82.41 (13)	O27—Na3—O29 ^{vii}	92.51 (16)
O11—W3—O8	87.05 (13)	O28—Na3—O7 ^{iv}	158.61 (15)
O11—W3—O10	92.51 (14)	O29 ^{vii} —Na3—O7 ^{iv}	83.56 (13)
O11—W3—O13	94.39 (14)	O29 ^{vii} —Na3—O20 ^{vi}	156.19 (16)
O11—W3—O14	160.06 (14)	O29 ^{vii} —Na3—O28	76.17 (15)
O12—W3—O8	164.22 (13)	W1—O1—Na1	135.79 (18)
O12—W3—O10	97.08 (14)	W1—O2—W6 ⁱ	117.99 (16)
O12—W3—O11	103.95 (15)	W1—O4—W4	117.48 (16)
O12—W3—O13	103.07 (15)	W1—O4—Na2 ⁱⁱⁱ	116.67 (15)
O12—W3—O14	95.83 (14)	W4—O4—Na2 ⁱⁱⁱ	116.38 (15)

O13—W3—O8	87.06 (13)	W2—O5—W1	125.33 (15)
O13—W3—O10	156.41 (13)	W2—O5—W4	137.59 (16)
O13—W3—O14	83.45 (13)	W4—O5—W1	94.63 (12)
O14—W3—O8	73.06 (12)	W2—O6—W6 ⁱ	139.38 (17)
O4—W4—O5	73.85 (12)	W2—O7—Na3 ^{iv}	137.29 (18)
O4—W4—O11 ⁱ	81.97 (13)	W3—O8—W2	97.22 (12)
O11 ⁱ —W4—O5	77.76 (12)	W5—O8—W2	94.75 (11)
O15—W4—O4	92.61 (14)	W5—O8—W3	94.52 (12)
O15—W4—O5	96.44 (14)	W5—O9—W2	116.91 (15)
O15—W4—O11 ⁱ	172.94 (14)	W3—O10—W2	120.08 (15)
O15—W4—O17	96.69 (15)	W3—O11—W4 ⁱ	136.32 (18)
O16—W4—O4	96.54 (14)	W3—O12—Na1 ^{viii}	170.67 (19)
O16—W4—O5	160.85 (13)	W3—O13—W1 ⁱ	126.28 (15)
O16—W4—O11 ⁱ	84.60 (13)	W3—O13—W6	137.29 (16)
O16—W4—O15	100.58 (15)	W6—O13—W1 ⁱ	95.12 (12)
O16—W4—O17	99.46 (15)	W5—O14—W3	115.64 (16)
O17—W4—O4	159.66 (14)	W4—O17—W5	148.36 (18)
O17—W4—O5	87.12 (13)	W5—O18—Na3 ^{ix}	150.89 (19)
O17—W4—O11 ⁱ	87.11 (13)	W6—O19—W5	146.16 (18)
O9—W5—O8	76.09 (12)	W6—O20—Na3 ^{vi}	132.86 (18)
O9—W5—O14	93.33 (14)	W6—O21—Na1 ⁱ	135.54 (18)
O9—W5—O17	86.47 (14)	Na1—O22—H22A	122 (6)
O9—W5—O19	154.74 (14)	Na1—O22—H22B	114 (4)
O14—W5—O8	76.78 (12)	Na2—O22—Na1	98.47 (16)
O14—W5—O17	156.72 (13)	Na2—O22—H22A	104 (6)
O14—W5—O19	87.83 (14)	Na2—O22—H22B	113 (4)
O17—W5—O8	80.60 (12)	H22A—O22—H22B	104 (7)
O18—W5—O8	178.51 (14)	Na1—O23—Na2	93.15 (15)
O18—W5—O9	102.80 (15)	Na1—O23—H23A	113 (5)
O18—W5—O14	102.35 (15)	Na1—O23—H23B	138 (4)
O18—W5—O17	100.38 (14)	Na2—O23—H23A	88 (5)
O18—W5—O19	101.57 (15)	Na2—O23—H23B	113 (4)
O19—W5—O8	79.64 (12)	H23A—O23—H23B	101 (6)
O19—W5—O17	82.74 (13)	Na2—O24—H24A	126 (5)
O2 ⁱ —W6—O6 ⁱ	77.66 (13)	Na2—O24—H24B	98 (7)
O2 ⁱ —W6—O13	72.73 (12)	H24A—O24—H24B	101 (8)
O6 ⁱ —W6—O13	76.98 (12)	Na2—O25—H25	118 (5)
O19—W6—O2 ⁱ	156.59 (14)	C1—O25—Na2	130.9 (3)
O19—W6—O6 ⁱ	84.75 (13)	C1—O25—H25	107 (5)
O19—W6—O13	88.45 (13)	Na2—O26—H26	106 (4)
O20—W6—O2 ⁱ	94.54 (14)	C6—O26—Na2	150.9 (3)
O20—W6—O6 ⁱ	90.10 (14)	C6—O26—H26	100 (4)
O20—W6—O13	163.39 (13)	Na3—O27—H27	110 (6)
O20—W6—O19	100.87 (15)	C4—O27—Na3	131.5 (3)
O20—W6—O21	101.60 (16)	C4—O27—H27	112 (6)
O21—W6—O2 ⁱ	95.80 (14)	Na3—O28—H28A	110 (6)
O21—W6—O6 ⁱ	167.09 (14)	Na3—O28—H28B	107 (6)
O21—W6—O13	90.50 (14)	H28A—O28—H28B	108 (8)

O21—W6—O19	98.20 (15)	Na3 ^x —O29—H29	108 (5)
O1—Na1—Na2	130.03 (12)	C3—O29—Na3 ^x	107.8 (3)
O1—Na1—O21 ⁱ	87.94 (13)	C3—O29—H29	109 (5)
O1—Na1—O30B ⁱⁱⁱ	75.4 (4)	H1A—N1—H1B	109.5
O1—Na1—O30A ⁱⁱⁱ	82.9 (4)	H1A—N1—H1C	109.5
O12 ⁱⁱ —Na1—Na2	90.80 (11)	H1B—N1—H1C	109.5
O12 ⁱⁱ —Na1—O1	91.95 (13)	C2—N1—H1A	109.5
O12 ⁱⁱ —Na1—O21 ⁱ	169.87 (16)	C2—N1—H1B	109.5
O12 ⁱⁱ —Na1—O23	87.84 (14)	C2—N1—H1C	109.5
O12 ⁱⁱ —Na1—O30B ⁱⁱⁱ	106.9 (5)	H2A—N2—H2B	109.5
O12 ⁱⁱ —Na1—O30A ⁱⁱⁱ	94.4 (6)	H2A—N2—H2C	109.5
O21 ⁱ —Na1—Na2	81.56 (10)	H2B—N2—H2C	109.5
O21 ⁱ —Na1—O30B ⁱⁱⁱ	82.9 (6)	C5—N2—H2A	109.5
O21 ⁱ —Na1—O30A ⁱⁱⁱ	95.6 (6)	C5—N2—H2B	109.5
O22—Na1—Na2	40.33 (11)	C5—N2—H2C	109.5
O22—Na1—O1	89.70 (15)	O25—C1—H1D	109.6
O22—Na1—O12 ⁱⁱ	92.67 (15)	O25—C1—H1E	109.6
O22—Na1—O21 ⁱ	77.19 (14)	O25—C1—C2	110.2 (4)
O22—Na1—O23	84.94 (15)	H1D—C1—H1E	108.1
O22—Na1—O30B ⁱⁱⁱ	155.5 (6)	C2—C1—H1D	109.6
O22—Na1—O30A ⁱⁱⁱ	169.9 (6)	C2—C1—H1E	109.6
O23—Na1—Na2	44.61 (11)	N1—C2—C1	108.4 (4)
O23—Na1—O1	174.62 (17)	N1—C2—H2	109.3
O23—Na1—O21 ⁱ	91.32 (14)	N1—C2—C3	110.2 (4)
O23—Na1—O30B ⁱⁱⁱ	109.8 (4)	C1—C2—H2	109.3
O23—Na1—O30A ⁱⁱⁱ	102.5 (4)	C3—C2—C1	110.2 (4)
O30B ⁱⁱⁱ —Na1—Na2	149.3 (2)	C3—C2—H2	109.3
O30A ⁱⁱⁱ —Na1—Na2	146.5 (3)	O29—C3—C2	112.1 (4)
O30A ⁱⁱⁱ —Na1—O30B ⁱⁱⁱ	14.6 (2)	O29—C3—H3A	109.2
Na1—Na2—H22A	51.3 (16)	O29—C3—H3B	109.2
Na1—Na2—H23A	51.3 (15)	C2—C3—H3A	109.2
Na1—Na2—H24B	69.2 (17)	C2—C3—H3B	109.2
O4 ⁱⁱⁱ —Na2—Na1	80.92 (9)	H3A—C3—H3B	107.9
O4 ⁱⁱⁱ —Na2—H22A	72.6 (17)	O27—C4—H4A	110.5
O4 ⁱⁱⁱ —Na2—H23A	67.2 (16)	O27—C4—H4B	110.5
O4 ⁱⁱⁱ —Na2—H24B	150.2 (17)	O27—C4—C5	106.2 (4)
O22—Na2—Na1	41.20 (11)	H4A—C4—H4B	108.7
O22—Na2—O4 ⁱⁱⁱ	83.35 (14)	C5—C4—H4A	110.5
O22—Na2—H22A	15.0 (16)	C5—C4—H4B	110.5
O22—Na2—O23	83.43 (15)	N2—C5—C4	110.1 (4)
O22—Na2—H23A	90.4 (15)	N2—C5—H5	107.2
O22—Na2—O24	86.63 (15)	C4—C5—H5	107.2
O22—Na2—H24B	74.3 (17)	C6—C5—N2	111.0 (4)
O22—Na2—O25	100.64 (16)	C6—C5—C4	113.9 (4)
O22—Na2—O26	172.64 (19)	C6—C5—H5	107.2
H22A—Na2—H23A	95 (2)	O26—C6—C5	110.7 (4)
H22A—Na2—H24B	88 (2)	O26—C6—H6A	109.5
O23—Na2—Na1	42.24 (10)	O26—C6—H6B	109.5

O23—Na2—O4 ⁱⁱⁱ	82.65 (13)	C5—C6—H6A	109.5
O23—Na2—H22A	92.4 (16)	C5—C6—H6B	109.5
O23—Na2—H23A	17.7 (15)	H6A—C6—H6B	108.1
O23—Na2—H24B	75.5 (17)	O30A—O30B—Na1 ⁱⁱⁱ	60.3 (18)
H23A—Na2—H24B	93 (2)	O30B—O30A—Na1 ⁱⁱⁱ	105 (2)
Na2—O25—C1—C2	-81.3 (5)	O11 ⁱ —W4—O17—W5	-70.5 (3)
Na2—O26—C6—C5	-28.2 (9)	O12—W3—O11—W4 ⁱ	-55.6 (3)
Na3—O27—C4—C5	-70.6 (6)	O12—W3—O13—W1 ⁱ	60.5 (2)
Na3 ^x —O29—C3—C2	-169.3 (3)	O12—W3—O13—W6	-136.2 (2)
O2—W1—O1—Na1	-25.9 (3)	O13 ⁱ —W1—O1—Na1	46.8 (3)
O2 ⁱ —W6—O19—W5	47.2 (6)	O13—W3—O11—W4 ⁱ	49.1 (3)
O2 ⁱ —W6—O20—Na3 ^{vi}	-50.9 (2)	O13—W6—O19—W5	11.3 (3)
O2 ⁱ —W6—O21—Na1 ⁱ	-30.6 (3)	O13—W6—O20—Na3 ^{vi}	-90.0 (5)
O3—W1—O1—Na1	-125.2 (3)	O13—W6—O21—Na1 ⁱ	42.1 (3)
O4—W1—O1—Na1	131.8 (2)	O14—W3—O11—W4 ⁱ	132.0 (3)
O4—W4—O17—W5	-13.1 (6)	O14—W3—O13—W1 ⁱ	155.0 (2)
O5—W1—O1—Na1	82.1 (6)	O14—W3—O13—W6	-41.7 (2)
O5—W2—O6—W6 ⁱ	-42.3 (3)	O14—W5—O9—W2	-76.39 (18)
O5—W2—O7—Na3 ^{iv}	-150.8 (2)	O14—W5—O18—Na3 ^{ix}	-153.2 (4)
O5—W4—O17—W5	7.4 (3)	O15—W4—O17—W5	103.5 (3)
O6—W2—O7—Na3 ^{iv}	109.9 (2)	O16—W4—O17—W5	-154.6 (3)
O6 ⁱ —W6—O19—W5	88.4 (3)	O17—W5—O9—W2	80.29 (17)
O6 ⁱ —W6—O20—Na3 ^{vi}	-128.5 (2)	O17—W5—O14—W3	-13.9 (4)
O6 ⁱ —W6—O21—Na1 ⁱ	28.1 (8)	O17—W5—O18—Na3 ^{ix}	31.8 (4)
O7—W2—O6—W6 ⁱ	64.3 (3)	O18—W5—O9—W2	-179.85 (17)
O8—W2—O6—W6 ⁱ	-128.0 (3)	O18—W5—O14—W3	178.84 (17)
O8—W2—O7—Na3 ^{iv}	-23.3 (6)	O19—W5—O9—W2	15.6 (4)
O8—W3—O11—W4 ⁱ	135.9 (2)	O19—W5—O14—W3	-79.79 (17)
O8—W3—O13—W1 ⁱ	-131.77 (19)	O19—W5—O18—Na3 ^{ix}	116.5 (4)
O8—W3—O13—W6	31.6 (2)	O19—W6—O20—Na3 ^{vi}	146.9 (2)
O8—W5—O9—W2	-0.89 (15)	O19—W6—O21—Na1 ⁱ	130.6 (2)
O8—W5—O14—W3	0.08 (15)	O20—W6—O19—W5	177.5 (3)
O9—W2—O6—W6 ⁱ	-131.0 (3)	O20—W6—O21—Na1 ⁱ	-126.5 (3)
O9—W2—O7—Na3 ^{iv}	-65.0 (3)	O21—W6—O19—W5	-79.0 (3)
O9—W5—O14—W3	74.95 (18)	O21—W6—O20—Na3 ^{vi}	46.0 (3)
O9—W5—O18—Na3 ^{ix}	-56.9 (4)	O25—C1—C2—N1	-54.2 (5)
O10—W2—O6—W6 ⁱ	161.4 (3)	O25—C1—C2—C3	-174.9 (4)
O10—W2—O7—Na3 ^{iv}	16.9 (3)	O27—C4—C5—N2	163.1 (4)
O10—W3—O11—W4 ⁱ	-153.5 (2)	O27—C4—C5—C6	-71.6 (5)
O10—W3—O13—W1 ⁱ	-151.6 (2)	N1—C2—C3—O29	63.4 (5)
O10—W3—O13—W6	11.8 (5)	N2—C5—C6—O26	61.3 (5)
O11—W3—O13—W1 ⁱ	-45.0 (2)	C1—C2—C3—O29	-177.0 (4)
O11—W3—O13—W6	118.4 (2)	C4—C5—C6—O26	-63.6 (6)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O22—H22A \cdots O1 ⁱⁱⁱ	0.67 (7)	2.11 (7)	2.757 (5)	163 (8)
O23—H23A \cdots O16 ⁱⁱⁱ	0.78 (7)	2.04 (7)	2.802 (5)	165 (7)
O23—H23B \cdots O16 ^{vii}	0.91 (7)	1.91 (7)	2.811 (5)	175 (6)
O25—H25 \cdots O3 ^{xi}	0.73 (6)	2.05 (7)	2.761 (5)	164 (7)
O26—H26 \cdots O15 ⁱⁱⁱ	0.88 (7)	1.88 (7)	2.730 (5)	160 (6)
O27—H27 \cdots O2 ^{xi}	0.67 (7)	2.04 (7)	2.696 (5)	165 (8)
O28—H28A \cdots O21 ^{vi}	0.75 (8)	2.01 (8)	2.735 (6)	160 (8)
O29—H29 \cdots O10 ⁱ	0.81 (6)	1.91 (7)	2.692 (5)	163 (6)
N1—H1B \cdots O15 ^{xi}	0.91	1.82	2.723 (5)	170
N2—H2B \cdots O24	0.91	1.96	2.867 (6)	172

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