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Crystal structure of disodium 2-amino-6-oxo-6,7-dihydro-1*H*-purine-1,7-diide heptahydrate

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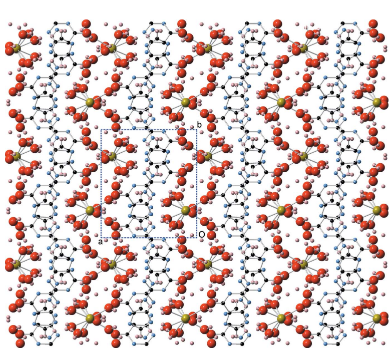
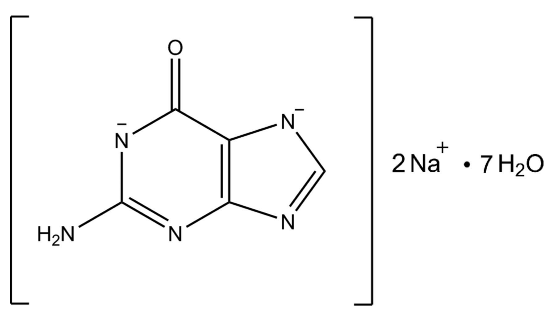


In the title compound, disodium 2-amino-6-oxo-6,7-dihydro-1*H*-purine-1,7-diide heptahydrate, $2Na^{+}\cdot C_5H_3N_5O^{2-}\cdot 7H_2O$, the structure is composed of alternating (100) layers of guanine molecules and hydrated Na^{+} ions. Within the guanine layer, the molecules are arranged in centrosymmetric pairs, with a partial overlap between the guanine rings. In this compound, guanine exists as the amino–keto tautomer from which deprotonation from N_1 and N_7 has occurred (purine numbering). There are no direct interactions between the Na^{+} cations and the guanine anions. Guanine molecules are linked to neighboring water molecules by O—H···N and O—H···O hydrogen bonds into a network structure.

1. Chemical context

Guanine is one of the five nucleic acids present in both DNA and RNA (Blackburn *et al.*, 2006), and is also found in its crystalline form in the integument of many animals as a light reflector (Land, 1972; Parker, 2000; Gur *et al.*, 2013, 2014). There are two known crystal structures of guanine; guanine monohydrate (Thewalt *et al.*, 1971) and anhydrous guanine (Guille & Clegg, 2006). In addition there are also a few known guanine salts (Broomhead, 1951; Wei, 1977; Iball & Wilson, 1965). The crystal structure of the title compound was obtained as a part of a study into controlling the crystal phase of guanine using recrystallization.



Cation, anion and radical formation among nucleic acids are thought to be important steps in DNA damage (Cooke *et al.*, 2003; Kasai, 1997). For that reason, protonation and deprotonation of nucleic acids and their role in processes like mutation has been widely studied both theoretically and experimentally. It is thought that the most prominent site for this kind of damage will be guanine because it has the lowest oxidation potential among the four DNA bases (Burrows & Muller, 1998; Steenken & Jovanovic, 1997). As a result, even initially different oxidized species may eventually migrate to guanine. Therefore, DNA damage is predicted to be produced at this site (Melvin *et al.*, 1995). The crystal structure of the

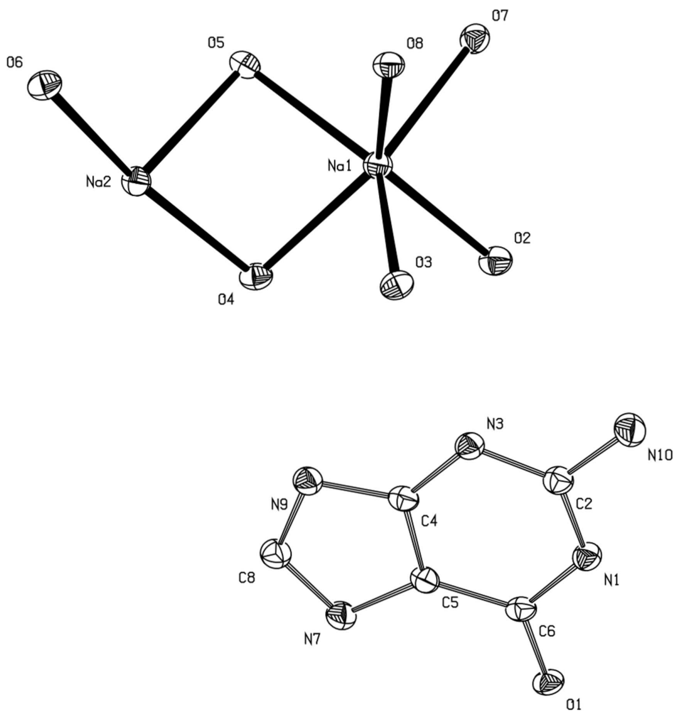


Figure 1
A displacement ellipsoid plot of the asymmetric unit drawn at the 50% probability level. H atoms have been omitted for clarity.

deprotonated guanine presented in this report may provide information about the deprotonated oxidized guanine state and its interactions with the neighboring water molecules.

2. Structural commentary

In the structure of the title compound, the asymmetric unit is composed of a guanine anion, two sodium counter-ions and seven water molecules (Fig. 1). In this compound, guanine exists as the amino–keto tautomer, the guanine molecules are doubly negatively charged, as a result of the deprotonation from N1 and N7 (purine numbering) that occurred due to the alkaline conditions of the solution from which recrystallization took place. There are no direct interactions between the Na^+ cations and the guanine anions.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N9^{i}	0.84 (3)	1.97 (3)	2.7875 (19)	168 (3)
O2—H2B···N3	0.89 (3)	2.08 (3)	2.9582 (19)	167 (2)
O3—H3A···O5^{ii}	0.87 (3)	2.08 (3)	2.9200 (18)	163 (3)
O3—H3B···N3	0.87 (3)	1.95 (3)	2.8038 (18)	166 (3)
O4—H4A···N1^{iii}	0.85 (3)	1.96 (3)	2.8093 (19)	177 (3)
O4—H4B···N9	0.85 (3)	2.14 (3)	2.9866 (19)	176 (2)
O5—H5C···O1^{iii}	0.81 (3)	1.96 (3)	2.7581 (18)	168 (3)
O6—H6A···O2^{iv}	0.79 (3)	2.02 (3)	2.7938 (19)	167 (3)
O6—H6B···N7^{v}	0.90 (3)	2.01 (3)	2.909 (2)	173 (2)
O7—H7A···O1^{v}	0.88 (3)	1.95 (3)	2.7867 (17)	160 (3)
O7—H7B···O3	0.85 (3)	1.92 (3)	2.7608 (18)	168 (3)
O8—H8A···O1^{iii}	0.84 (3)	1.99 (3)	2.8303 (17)	171 (3)
O8—H8B···N7^{vi}	0.82 (3)	1.98 (3)	2.7938 (19)	171 (3)
O5—H5D···O1^{vii}	0.78 (3)	2.02 (3)	2.7835 (17)	164 (3)

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

3. Supramolecular features

The structure is composed of alternating (100) layers of guanine molecules and hydrated Na^+ Ions (Fig. 2). Within the guanine layer, the molecules are arranged in centrosymmetric pairs, in which a partial overlap between the guanine rings is present. The distances between the overlapping atoms C_2–$N_3{}^{i}$ and C_4–$N_{10}{}^{i}$ are 3.415 (2) and 3.460 (2) Å, respectively [symmetry code: (i) = $1-x, 1-y, 1-z$]. The two molecules are offset presumably to separate the charged N^- ions of the two molecules and at the same time provide van der Waals contacts between the two rings. In most known guanine crystal structures, neighboring guanine molecules form hydrogen bonds that result in flat layers of guanine molecules, between which stacking interactions are present. Such layers are not

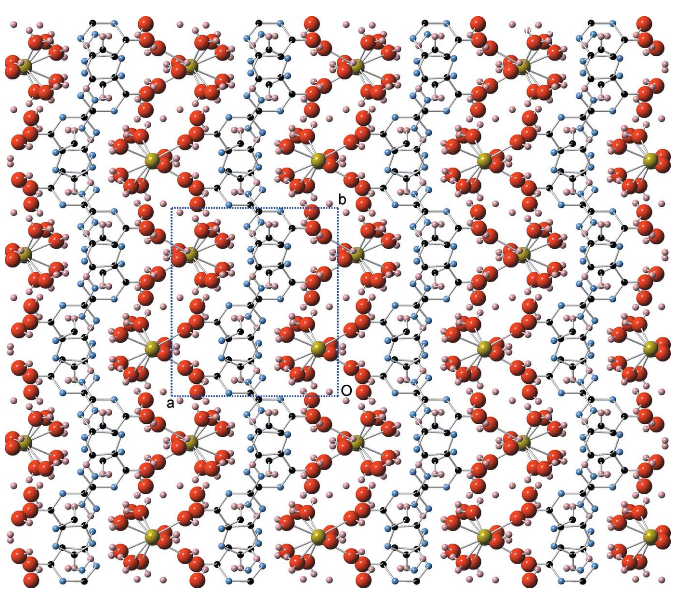


Figure 2
The crystal structure viewed down the c axis, showing the alternating layers of guanine molecules and hydrated sodium ions.

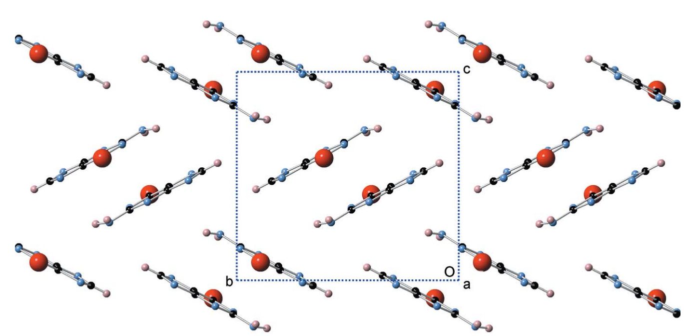


Figure 3
A view down the a axis showing the herringbone crystal packing motif, including edge-to-face interactions between the guanine dimers.

Table 2
Experimental details.

Crystal data	
Chemical formula	$2Na^+ \cdot C_5H_3N_5O^{2-} \cdot 7H_2O$
M_r	321.21
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, b, c (Å)	10.5520 (2), 11.6936 (3), 11.1938 (2)
β (°)	101.5758 (13)
V (Å^3)	1353.12 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.20
Crystal size (mm)	0.30 × 0.10 × 0.05
Data collection	
Diffractometer	Nonius KappaCCD
Absorption correction	Multi-scan (*DENZO-SMN*; Otwinowski & Minor, 2006)
T_{min}, T_{max}	0.977, 0.990
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6648, 3931, 2981
R_{int}	0.019
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.704
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.147, 1.07
No. of reflections	3931
No. of parameters	248
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.57, −0.39

Computer programs: *COLLECT* (Nonius, 1998), *DENZO-SMN* (Otwinowski & Minor, 2006), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *PLATON* (Spek, 2009), *CrystalMaker* (CrystalMaker, 2010) and *publCIF* (Westrip, 2010).

present in the structure of the title compound. Instead, the guanine molecules form O—H···N and O—H···O hydrogen bonds with the neighboring water molecules (Table 1), satisfying all guanine donors and acceptors with the exception of the NH_2 amine group, which surprisingly does not seem to participate in any hydrogen bonding, and is not within hydrogen-bonding distance of any hydrogen acceptors. In addition, the guanine molecules form dimers that have an edge-to-face type orientation, resulting in the observed herringbone crystal packing motif with a dihedral angle of 123.917 (17)° (Fig. 3).

4. Synthesis and crystallization

Disodium 2-amino-6-oxo-6,7-dihydro-1*H*-purine-1,7-diide heptahydrate was prepared by dissolving 0.1 g guanine (powder Sigma–Aldrich) in 5 ml NaOH 1 *N* (pH 14). The solution was then filtered using a PVDF filter (0.22 µm), and 0.1 ml of NaOH 1 *N* was added to the solution to ensure that all of the guanine was dissolved. The solution was then kept for 10 days under an IR lamp using 15 min. cycles (on/off) while open to the atmosphere. Large 3mm crystals were extracted from the suspension, broken to a suitable size and subjected to single crystal X-ray diffraction.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were refined freely with the exception of C8-bound H atom that was placed in a calculated position and refined in riding mode.

Acknowledgements

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Crystal structure of disodium 2-amino-6-oxo-6,7-dihydro-1*H*-purine-1,7-diide heptahydrate

Dvir Gur and Linda J. W. Shimon

Computing details

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 2006); data reduction: *DENZO-SMN* (Otwinowski & Minor, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009) and *CrystalMaker* (*CrystalMaker*, 2010); software used to prepare material for publication: *SHELXL2013* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

Disodium 2-amino-6-oxo-6,7-dihydro-1*H*-purine-1,7-diide heptahydrate

Crystal data

$2Na^+\cdot C_5H_3N_5O^{2-}\cdot 7H_2O$
$M_r = 321.21$
Monoclinic, $P2_1/c$
$a = 10.5520$ (2) Å
$b = 11.6936$ (3) Å
$c = 11.1938$ (2) Å
$\beta = 101.5758$ (13)°
$V = 1353.12$ (5) Å^3
$Z = 4$
$F(000) = 672$
$D_x = 1.577$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3810 reflections
$\theta = 2.6$–$30.0°$
$\mu = 0.20$ mm^{-1}
$T = 120$ K
Plate, colourless
0.30 × 0.10 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: sealed tube
φ and ω scans
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 2006)
$T_{min} = 0.977$, $T_{max} = 0.990$
6648 measured reflections
3931 independent reflections
2981 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 30.0°$, $\theta_{min} = 3.7°$
$h = -14\rightarrow 14$
$k = -12\rightarrow 16$
$l = -15\rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.147$
$S = 1.07$
3931 reflections
248 parameters
0 restraints
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0862P)^2 + 0.5094P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.65524 (13)	0.48287 (13)	0.34832 (13)	0.0172 (3)
C2	0.52557 (16)	0.48307 (15)	0.33930 (15)	0.0175 (3)
N3	0.45252 (13)	0.40241 (13)	0.37665 (13)	0.0170 (3)
C4	0.52344 (15)	0.31289 (15)	0.43267 (14)	0.0158 (3)
C5	0.65775 (15)	0.30526 (15)	0.44968 (14)	0.0161 (3)
C6	0.72597 (15)	0.39376 (14)	0.40446 (14)	0.0156 (3)
N7	0.69798 (13)	0.20349 (13)	0.50957 (13)	0.0186 (3)
C8	0.58601 (16)	0.15733 (16)	0.52393 (16)	0.0193 (3)
H8	0.585 (2)	0.087 (2)	0.563 (2)	0.023*
N9	0.47610 (13)	0.21804 (13)	0.47962 (13)	0.0178 (3)
N10	0.46123 (16)	0.57498 (15)	0.27867 (15)	0.0235 (3)
H10A	0.378 (3)	0.585 (3)	0.290 (3)	0.057 (9)*
H10B	0.513 (3)	0.636 (3)	0.271 (3)	0.048 (8)*
O1	0.85043 (11)	0.39326 (11)	0.41331 (10)	0.0171 (3)
Na1	0.11549 (6)	0.25431 (6)	0.18356 (6)	0.01689 (17)
Na2	0.04858 (6)	0.04502 (6)	0.37257 (6)	0.01704 (17)
O2	0.30698 (12)	0.32592 (11)	0.13607 (12)	0.0194 (3)
H2A	0.355 (3)	0.303 (3)	0.090 (3)	0.043 (8)*
H2B	0.362 (3)	0.346 (2)	0.204 (2)	0.031 (6)*
O3	0.18432 (12)	0.38036 (11)	0.35525 (11)	0.0186 (3)
H3A	0.143 (3)	0.445 (3)	0.352 (3)	0.048 (8)*
H3B	0.267 (3)	0.391 (3)	0.374 (3)	0.042 (7)*
O4	0.24329 (12)	0.11485 (11)	0.32009 (11)	0.0177 (3)
H4A	0.276 (3)	0.075 (2)	0.271 (3)	0.035 (7)*
H4B	0.307 (3)	0.146 (2)	0.367 (2)	0.032 (7)*
O5	−0.00208 (13)	0.06838 (11)	0.14535 (11)	0.0174 (3)
H5C	0.050 (3)	0.024 (3)	0.128 (3)	0.042 (8)*
H5D	−0.051 (3)	0.089 (3)	0.087 (3)	0.040 (7)*
O6	−0.15505 (12)	0.02121 (12)	0.42404 (11)	0.0200 (3)
H6A	−0.207 (3)	−0.027 (3)	0.401 (3)	0.037 (7)*
H6B	−0.203 (3)	0.079 (2)	0.444 (2)	0.037 (7)*
O7	0.04292 (13)	0.22690 (11)	0.46296 (11)	0.0180 (3)
H7A	−0.030 (3)	0.265 (3)	0.447 (3)	0.041 (7)*
H7B	0.094 (3)	0.274 (2)	0.440 (3)	0.034 (7)*
O8	0.08223 (12)	−0.14985 (11)	0.31485 (11)	0.0175 (3)
H8A	0.108 (2)	−0.143 (2)	0.249 (3)	0.033 (7)*
H8B	0.145 (3)	−0.173 (2)	0.365 (3)	0.034 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0152 (6)	0.0196 (7)	0.0171 (6)	−0.0014 (5)	0.0042 (5)	0.0002 (5)
C2	0.0158 (7)	0.0205 (8)	0.0162 (7)	0.0002 (6)	0.0034 (6)	−0.0002 (6)
N3	0.0139 (6)	0.0197 (7)	0.0172 (6)	−0.0007 (5)	0.0030 (5)	0.0006 (5)
C4	0.0130 (7)	0.0214 (8)	0.0127 (7)	0.0011 (6)	0.0023 (5)	−0.0006 (6)
C5	0.0141 (7)	0.0201 (8)	0.0140 (7)	0.0015 (6)	0.0024 (5)	0.0016 (6)
C6	0.0131 (7)	0.0207 (8)	0.0128 (7)	−0.0008 (6)	0.0021 (5)	−0.0024 (6)
N7	0.0154 (6)	0.0205 (7)	0.0194 (7)	0.0015 (5)	0.0022 (5)	0.0026 (5)
C8	0.0163 (8)	0.0220 (9)	0.0196 (8)	0.0012 (6)	0.0033 (6)	0.0020 (6)
N9	0.0154 (6)	0.0209 (7)	0.0169 (6)	0.0000 (5)	0.0030 (5)	0.0016 (5)
N10	0.0180 (7)	0.0243 (8)	0.0282 (8)	0.0031 (6)	0.0045 (6)	0.0072 (6)
O1	0.0106 (5)	0.0232 (6)	0.0177 (5)	−0.0012 (4)	0.0034 (4)	−0.0005 (4)
Na1	0.0157 (3)	0.0191 (4)	0.0157 (3)	−0.0004 (2)	0.0028 (2)	0.0002 (2)
Na2	0.0166 (3)	0.0193 (3)	0.0158 (3)	0.0003 (2)	0.0045 (2)	−0.0001 (2)
O2	0.0159 (6)	0.0261 (7)	0.0167 (6)	−0.0009 (5)	0.0045 (5)	−0.0018 (5)
O3	0.0135 (6)	0.0207 (6)	0.0223 (6)	0.0008 (5)	0.0047 (4)	−0.0024 (5)
O4	0.0134 (5)	0.0220 (6)	0.0176 (6)	0.0002 (5)	0.0030 (4)	−0.0028 (5)
O5	0.0166 (6)	0.0202 (6)	0.0146 (5)	0.0028 (5)	0.0013 (4)	0.0013 (5)
O6	0.0159 (6)	0.0230 (7)	0.0213 (6)	−0.0008 (5)	0.0043 (5)	−0.0027 (5)
O7	0.0171 (6)	0.0198 (6)	0.0177 (6)	0.0017 (5)	0.0049 (4)	0.0003 (4)
O8	0.0147 (6)	0.0229 (6)	0.0147 (5)	0.0016 (5)	0.0022 (4)	0.0012 (4)

Geometric parameters (Å, º)

N1—C2	1.352 (2)	Na2—O7	2.3612 (14)
N1—C6	1.360 (2)	Na2—O4	2.3912 (14)
C2—N3	1.337 (2)	Na2—O8	2.4142 (15)
C2—N10	1.375 (2)	Na2—O6^{iii}	2.4534 (14)
N3—C4	1.364 (2)	Na2—O5	2.5067 (14)
C4—N9	1.364 (2)	Na2—Na2^{iii}	3.3871 (13)
C4—C5	1.394 (2)	Na2—Na1^{iv}	3.8095 (9)
C5—N7	1.390 (2)	Na2—Na1^{v}	4.1397 (9)
C5—C6	1.411 (2)	O2—H2A	0.84 (3)
C6—O1	1.2970 (19)	O2—H2B	0.89 (3)
N7—C8	1.338 (2)	O3—H3A	0.87 (3)
C8—N9	1.365 (2)	O3—H3B	0.87 (3)
C8—H8	0.94 (2)	O4—H4A	0.85 (3)
N10—H10A	0.91 (3)	O4—H4B	0.85 (3)
N10—H10B	0.91 (3)	O5—H5C	0.81 (3)
Na1—O2	2.3447 (14)	O5—H5D	0.78 (3)
Na1—O8^{i}	2.3715 (14)	O6—Na2^{iii}	2.4534 (14)
Na1—O3	2.4153 (14)	O6—H6A	0.79 (3)
Na1—O7^{ii}	2.4440 (14)	O6—H6B	0.90 (3)
Na1—O4	2.4467 (14)	O7—Na1^{v}	2.4440 (14)
Na1—O5	2.4972 (14)	O7—H7A	0.88 (3)
Na1—Na2	3.4006 (9)	O7—H7B	0.85 (3)

Na1—Na2^{i}	3.8095 (9)	O8—Na1^{iv}	2.3716 (14)
Na1—Na2^{ii}	4.1397 (9)	O8—H8A	0.84 (3)
Na2—O6	2.3502 (14)	O8—H8B	0.82 (3)
C2—N1—C6	119.30 (14)	O4—Na2—O5	74.47 (4)
N3—C2—N1	127.87 (15)	O8—Na2—O5	81.04 (5)
N3—C2—N10	116.55 (15)	O6^{iii}—Na2—O5	160.17 (5)
N1—C2—N10	115.49 (15)	O6—Na2—Na2^{iii}	46.41 (3)
C2—N3—C4	112.80 (14)	O7—Na2—Na2^{iii}	83.17 (4)
N3—C4—N9	126.26 (14)	O4—Na2—Na2^{iii}	137.16 (5)
N3—C4—C5	124.10 (15)	O8—Na2—Na2^{iii}	91.01 (4)
N9—C4—C5	109.64 (14)	O6^{iii}—Na2—Na2^{iii}	43.93 (3)
N7—C5—C4	108.93 (14)	O5—Na2—Na2^{iii}	148.16 (5)
N7—C5—C6	132.26 (14)	O6—Na2—Na1	123.50 (4)
C4—C5—C6	118.80 (15)	O7—Na2—Na1	69.18 (4)
O1—C6—N1	119.48 (15)	O4—Na2—Na1	46.01 (3)
O1—C6—C5	123.43 (15)	O8—Na2—Na1	116.92 (4)
N1—C6—C5	117.09 (14)	O6^{iii}—Na2—Na1	133.71 (4)
C8—N7—C5	102.23 (14)	O5—Na2—Na1	47.07 (3)
N7—C8—N9	116.93 (16)	Na2^{iii}—Na2—Na1	152.07 (3)
N7—C8—H8	120.6 (14)	O6—Na2—Na1^{iv}	61.78 (4)
N9—C8—H8	122.5 (14)	O7—Na2—Na1^{iv}	145.56 (4)
C4—N9—C8	102.28 (13)	O4—Na2—Na1^{iv}	130.44 (4)
C2—N10—H10A	115 (2)	O8—Na2—Na1^{iv}	36.86 (3)
C2—N10—H10B	114.3 (19)	O6^{iii}—Na2—Na1^{iv}	88.83 (4)
H10A—N10—H10B	121 (3)	O5—Na2—Na1^{iv}	86.14 (4)
O2—Na1—O8^{i}	129.23 (5)	Na2^{iii}—Na2—Na1^{iv}	69.95 (2)
O2—Na1—O3	80.05 (5)	Na1—Na2—Na1^{iv}	133.15 (2)
O8^{i}—Na1—O3	80.22 (5)	O6—Na2—Na1^{v}	82.59 (4)
O2—Na1—O7^{ii}	81.28 (5)	O7—Na2—Na1^{v}	31.10 (3)
O8^{i}—Na1—O7^{ii}	82.37 (5)	O4—Na2—Na1^{v}	90.17 (4)
O3—Na1—O7^{ii}	137.08 (5)	O8—Na2—Na1^{v}	138.65 (4)
O2—Na1—O4	89.29 (5)	O6^{iii}—Na2—Na1^{v}	55.22 (4)
O8^{i}—Na1—O4	133.29 (5)	O5—Na2—Na1^{v}	139.21 (4)
O3—Na1—O4	82.51 (5)	Na2^{iii}—Na2—Na1^{v}	59.82 (2)
O7^{ii}—Na1—O4	135.46 (5)	Na1—Na2—Na1^{v}	95.369 (16)
O2—Na1—O5	133.89 (5)	Na1^{iv}—Na2—Na1^{v}	129.771 (19)
O8^{i}—Na1—O5	90.29 (5)	Na1—O2—H2A	133 (2)
O3—Na1—O5	136.66 (5)	Na1—O2—H2B	110.3 (16)
O7^{ii}—Na1—O5	82.01 (5)	H2A—O2—H2B	104 (2)
O4—Na1—O5	73.70 (5)	Na1—O3—H3A	115 (2)
O2—Na1—Na2	133.95 (4)	Na1—O3—H3B	114.2 (19)
O8^{i}—Na1—Na2	92.44 (4)	H3A—O3—H3B	110 (3)
O3—Na1—Na2	90.63 (4)	Na2—O4—Na1	89.31 (5)
O7^{ii}—Na1—Na2	129.14 (4)	Na2—O4—H4A	117.3 (18)
O4—Na1—Na2	44.68 (3)	Na1—O4—H4A	101.7 (18)
O5—Na1—Na2	47.31 (3)	Na2—O4—H4B	127.2 (17)
O2—Na1—Na2^{i}	91.59 (4)	Na1—O4—H4B	112.0 (18)

O8^{i}—Na1—Na2^{i}	37.64 (3)	H4A—O4—H4B	105 (2)
O3—Na1—Na2^{i}	68.95 (4)	Na1—O5—Na2	85.62 (4)
O7^{ii}—Na1—Na2^{i}	73.34 (4)	Na1—O5—H5C	105 (2)
O4—Na1—Na2^{i}	150.82 (4)	Na2—O5—H5C	99 (2)
O5—Na1—Na2^{i}	123.71 (4)	Na1—O5—H5D	96 (2)
Na2—Na1—Na2^{i}	126.913 (19)	Na2—O5—H5D	148 (2)
O2—Na1—Na2^{ii}	67.43 (4)	H5C—O5—H5D	111 (3)
O8^{i}—Na1—Na2^{ii}	74.73 (4)	Na2—O6—Na2^{iii}	89.65 (5)
O3—Na1—Na2^{ii}	107.25 (4)	Na2—O6—H6A	128 (2)
O7^{ii}—Na1—Na2^{ii}	29.94 (3)	Na2^{iii}—O6—H6A	104 (2)
O4—Na1—Na2^{ii}	151.97 (4)	Na2—O6—H6B	124.0 (17)
O5—Na1—Na2^{ii}	110.76 (4)	Na2^{iii}—O6—H6B	100.6 (17)
Na2—Na1—Na2^{ii}	155.43 (3)	H6A—O6—H6B	103 (3)
Na2^{i}—Na1—Na2^{ii}	50.230 (19)	Na2—O7—Na1^{v}	118.96 (6)
O6—Na2—O7	84.18 (5)	Na2—O7—H7A	117.7 (19)
O6—Na2—O4	166.81 (6)	Na1^{v}—O7—H7A	104.2 (19)
O7—Na2—O4	83.95 (5)	Na2—O7—H7B	112.8 (18)
O6—Na2—O8	98.31 (5)	Na1^{v}—O7—H7B	99.0 (18)
O7—Na2—O8	169.31 (5)	H7A—O7—H7B	101 (3)
O4—Na2—O8	94.41 (5)	Na1^{iv}—O8—Na2	105.50 (5)
O6—Na2—O6^{iii}	90.35 (5)	Na1^{iv}—O8—H8A	119.6 (18)
O7—Na2—O6^{iii}	86.16 (5)	Na2—O8—H8A	103.7 (19)
O4—Na2—O6^{iii}	94.57 (5)	Na1^{iv}—O8—H8B	115.2 (18)
O8—Na2—O6^{iii}	83.44 (5)	Na2—O8—H8B	105.6 (19)
O6—Na2—O5	104.03 (5)	H8A—O8—H8B	106 (3)
O7—Na2—O5	108.55 (5)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N9^{ii}	0.84 (3)	1.97 (3)	2.7875 (19)	168 (3)
O2—H2*B*···N3	0.89 (3)	2.08 (3)	2.9582 (19)	167 (2)
O3—H3*A*···O5^{i}	0.87 (3)	2.08 (3)	2.9200 (18)	163 (3)
O3—H3*B*···N3	0.87 (3)	1.95 (3)	2.8038 (18)	166 (3)
O4—H4*A*···N1^{vi}	0.85 (3)	1.96 (3)	2.8093 (19)	177 (3)
O4—H4*B*···N9	0.85 (3)	2.14 (3)	2.9866 (19)	176 (2)
O5—H5*C*···O1^{vi}	0.81 (3)	1.96 (3)	2.7581 (18)	168 (3)
O6—H6*A*···O2^{iv}	0.79 (3)	2.02 (3)	2.7938 (19)	167 (3)
O6—H6*B*···N7^{vii}	0.90 (3)	2.01 (3)	2.909 (2)	173 (2)
O7—H7*A*···O1^{vii}	0.88 (3)	1.95 (3)	2.7867 (17)	160 (3)
O7—H7*B*···O3	0.85 (3)	1.92 (3)	2.7608 (18)	168 (3)
O8—H8*A*···O1^{vi}	0.84 (3)	1.99 (3)	2.8303 (17)	171 (3)
O8—H8*B*···N7^{viii}	0.82 (3)	1.98 (3)	2.7938 (19)	171 (3)
O5—H5*D*···O1^{ix}	0.78 (3)	2.02 (3)	2.7835 (17)	164 (3)

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*−1/2; (iv) −*x*, *y*−1/2, −*z*+1/2; (vi) −*x*+1, *y*−1/2, −*z*+1/2; (vii) *x*−1, *y*, *z*; (viii) −*x*+1, −*y*, −*z*+1; (ix) *x*−1, −*y*+1/2, *z*−1/2.