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Dimethanolbis[*N*′-(3-pyridylmethylene)-benzohydrazide]sodium(I) iodide

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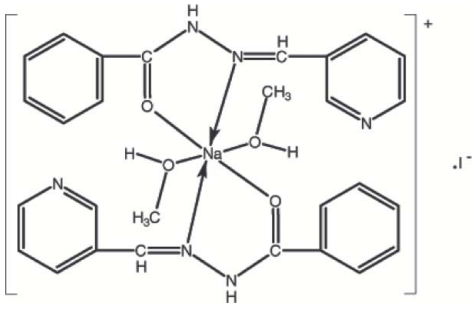
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.037; wR factor = 0.104; data-to-parameter ratio = 14.0.

The molecule of the title compound, $[Na(C_{13}H_{11}N_3O)_2(CH_3OH)_2]I$, is non-planar, with the Na atom chelated by the O atoms and the N atoms of two *N*′-(3-pyridylmethylene)benzohydrazide ligands and both O atoms of two methanol ligands. The asymmetric unit consists of one half-molecule. The Na atom is located on a crystallographic centre of inversion. The six-coordinate Na atom adopts a distorted octahedral coordination. In the crystal structure, intermolecular N—H···I and O—H···N hydrogen bonds link the molecules into a two-dimensional network.

Related literature

For general background, see: Lindoy *et al.* (1976). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$[Na(C_{13}H_{11}N_3O)_2(CH_4O)_2]I$
M_r = 664.47
Monoclinic, $P2_1/n$
a = 8.6078 (15) Å
b = 13.1842 (16) Å
c = 13.2508 (17) Å
β = 101.6540 (10)°
V = 1472.8 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.15 mm^{-1}
T = 298 (2) K
0.54 × 0.43 × 0.40 mm

Data collection

Bruker SMART CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.577, T_{max} = 0.657
7140 measured reflections
2586 independent reflections
1919 reflections with $I > 2\sigma(I)$
R_{int} = 0.078

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.104
S = 1.05
2586 reflections
185 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Na1—O1	2.294 (3)	Na1—N2	2.642 (3)
Na1—O2	2.344 (2)		
O1—Na1—O2	86.74 (10)	O2—Na1—N2	88.40 (9)
O1—Na1—N2	65.34 (9)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···I1^{i}	0.86	3.03	3.816 (3)	153
O2—H2···N3^{ii}	0.82	1.98	2.792 (5)	171

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2678).

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supporting information

Acta Cryst. (2009). E**65**, m38 [doi:10.1107/S1600536808040750]

Dimethanolbis[*N*′-(3-pyridylmethylene)benzohydrazide]sodium(I) iodide

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S1. Comment

Schiff bases have been known as effective ligands for metal ions in the preparation of dyes for many years, liquid crystals and powerful corrosion inhibitors. Furthermore, they are used in the mechanism of many biochemical processes (Lindoy *et al.*, 1976). We report here the synthesis and crystal structure of the title compound (I).

The molecular structure of (I) is shown in Fig.1. The values of the geometric parameters in (I) are normal (Allen *et al.*, 1987) (Table 1). In the crystal structure, there exist two intermolecular N—H···I and O—H···N hydrogen bonds (Table 2). As seen in Fig.2, the molecules are linked into two-dimensional network.

S2. Experimental

A mixture of *N*′-(3-pyridylmethylene)benzohydrazide (3 mmol) and sodium methoxide (3 mmol) and bismuth iodide(1 mmol) in absolute ethanol (15 ml) was heated under reflux with stirring for 5 h and then filtered.The resulting clear colourless solution was diffused diethyl ether vapor at room temperature for 16 days, after which large colourless block-shaped crystals of the title complex suitable for X-ray diffraction analysis were obtained.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model, with C—H = 0.96 Å (methylene) or 0.93 Å (aromatic),0.82 Å (hydroxyl) and U_{iso}(H) =1.2U_{eq}(C).

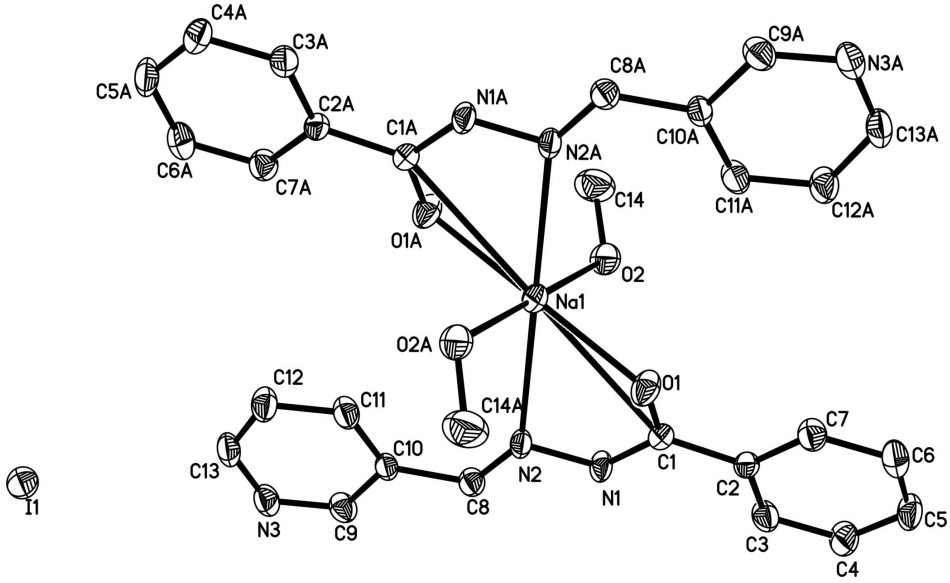

Figure 1

The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

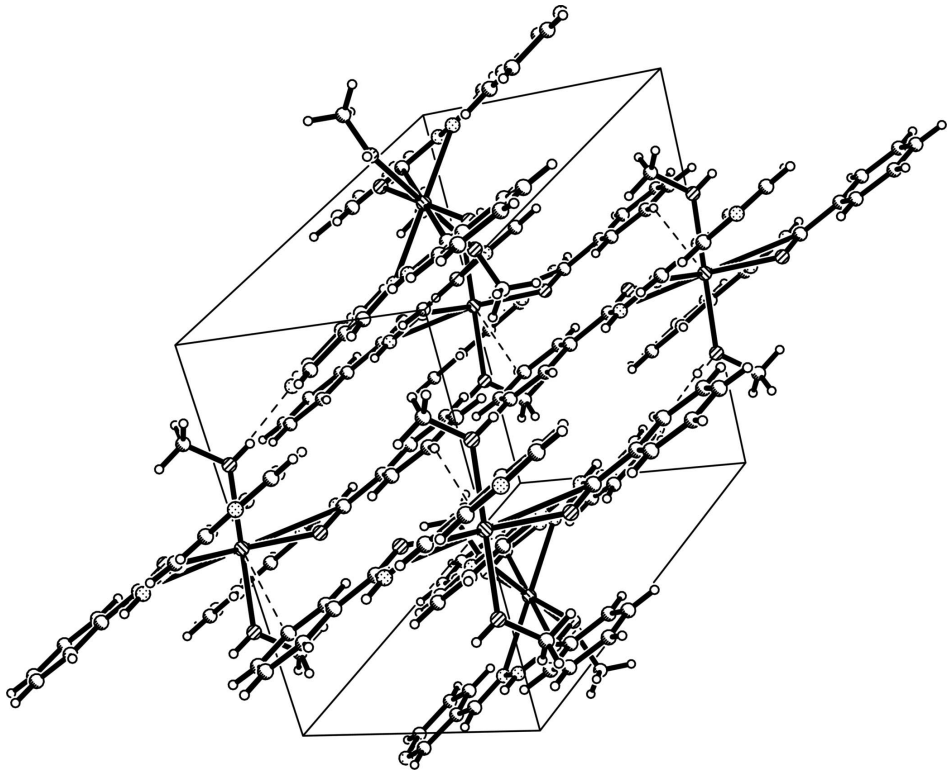

Figure 2

The crystal packing of the title compound, viewed approximately along the *c* axis.

Dimethanolbis[*N*′-(3-pyridylmethylene)benzohydrazide]sodium(I) iodide

Crystal data

$[Na(C_{13}H_{11}N_3O)_2(CH_4O)_2]I$
$M_r = 664.47$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.6078\ (15)$ Å
$b = 13.1842\ (16)$ Å
$c = 13.2508\ (17)$ Å
$\beta = 101.654\ (1)°$
$V = 1472.8\ (4)$ Å^3
$Z = 2$
$F(000) = 672.0$
$D_x = 1.494$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3137 reflections
$\theta = 2.2–27.2°$
$\mu = 1.15$ mm^{-1}
$T = 298$ K
Block, colourless
0.54 × 0.43 × 0.40 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.577$, $T_{max} = 0.657$
7140 measured reflections
2586 independent reflections
1919 reflections with $I > 2\sigma(I)$
$R_{int} = 0.078$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -10\rightarrow9$
$k = -15\rightarrow12$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.104$
$S = 1.05$
2586 reflections
185 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.028P)^2 + 0.7317P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.69$ e Å^{-3}
$\Delta\rho_{min} = -0.87$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0258 (18)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Na1	0.5000	0.5000	0.0000	0.0383 (5)
I1	0.0000	0.5000	0.5000	0.0439 (2)
N1	0.6395 (4)	0.2735 (2)	0.0500 (2)	0.0384 (8)
H1	0.6445	0.2085	0.0462	0.046*
N2	0.5180 (3)	0.3195 (2)	0.0883 (2)	0.0347 (7)
N3	0.0867 (4)	0.2404 (3)	0.2536 (3)	0.0470 (9)
O1	0.7451 (3)	0.42504 (19)	0.0231 (2)	0.0480 (7)
O2	0.4427 (3)	0.4186 (2)	−0.16052 (19)	0.0492 (7)
H2	0.4879	0.3694	−0.1793	0.074*
C1	0.7503 (4)	0.3326 (3)	0.0187 (3)	0.0336 (8)
C2	0.8797 (4)	0.2795 (3)	−0.0193 (3)	0.0319 (8)
C3	0.8939 (5)	0.1758 (3)	−0.0264 (3)	0.0455 (10)
H3	0.8179	0.1337	−0.0075	0.055*
C4	1.0197 (6)	0.1342 (3)	−0.0613 (3)	0.0564 (12)
H4	1.0281	0.0641	−0.0654	0.068*
C5	1.1334 (5)	0.1946 (4)	−0.0902 (3)	0.0526 (11)
H5	1.2187	0.1658	−0.1131	0.063*
C6	1.1193 (5)	0.2980 (3)	−0.0848 (3)	0.0498 (11)
H6	1.1948	0.3398	−0.1048	0.060*
C7	0.9930 (4)	0.3401 (3)	−0.0497 (3)	0.0424 (9)
H7	0.9842	0.4103	−0.0465	0.051*
C8	0.4330 (5)	0.2585 (3)	0.1286 (3)	0.0382 (9)
H8	0.4564	0.1895	0.1299	0.046*

C9	0.2103 (5)	0.2198 (3)	0.2102 (3)	0.0434 (10)
H9	0.2379	0.1522	0.2045	0.052*
C10	0.3003 (4)	0.2933 (3)	0.1730 (3)	0.0329 (8)
C11	0.2568 (5)	0.3933 (3)	0.1804 (3)	0.0439 (10)
H11	0.3129	0.4450	0.1561	0.053*
C12	0.1293 (5)	0.4150 (3)	0.2242 (3)	0.0487 (10)
H12	0.0978	0.4819	0.2299	0.058*
C13	0.0492 (5)	0.3378 (3)	0.2592 (3)	0.0471 (10)
H13	−0.0368	0.3542	0.2887	0.057*
C14	0.3307 (7)	0.4569 (4)	−0.2446 (3)	0.0740 (15)
H14A	0.2857	0.5185	−0.2245	0.111*
H14B	0.2481	0.4078	−0.2655	0.111*
H14C	0.3821	0.4705	−0.3011	0.111*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0414 (12)	0.0344 (12)	0.0424 (11)	0.0097 (9)	0.0161 (9)	−0.0003 (9)
I1	0.0536 (3)	0.0320 (3)	0.0480 (3)	0.00088 (16)	0.01502 (17)	−0.00036 (16)
N1	0.0429 (19)	0.0277 (16)	0.0514 (19)	0.0058 (14)	0.0258 (16)	−0.0028 (14)
N2	0.0336 (17)	0.0352 (17)	0.0395 (17)	0.0050 (14)	0.0178 (14)	−0.0020 (13)
N3	0.046 (2)	0.054 (2)	0.047 (2)	−0.0037 (17)	0.0229 (17)	0.0043 (16)
O1	0.0430 (16)	0.0313 (15)	0.0748 (19)	0.0031 (12)	0.0237 (14)	−0.0019 (13)
O2	0.0611 (19)	0.0450 (17)	0.0432 (15)	0.0100 (14)	0.0145 (14)	−0.0067 (13)
C1	0.033 (2)	0.033 (2)	0.0365 (19)	0.0057 (17)	0.0096 (16)	0.0026 (16)
C2	0.034 (2)	0.033 (2)	0.0294 (18)	0.0052 (17)	0.0082 (15)	−0.0014 (15)
C3	0.057 (3)	0.035 (2)	0.052 (2)	0.0053 (19)	0.028 (2)	0.0038 (18)
C4	0.067 (3)	0.044 (3)	0.067 (3)	0.018 (2)	0.035 (2)	0.002 (2)
C5	0.044 (3)	0.069 (3)	0.050 (3)	0.013 (2)	0.024 (2)	−0.006 (2)
C6	0.041 (2)	0.064 (3)	0.048 (2)	−0.006 (2)	0.020 (2)	−0.003 (2)
C7	0.042 (2)	0.040 (2)	0.047 (2)	−0.0017 (19)	0.0153 (18)	0.000 (2)
C8	0.044 (2)	0.032 (2)	0.042 (2)	0.0015 (17)	0.0164 (19)	0.0000 (16)
C9	0.053 (3)	0.037 (2)	0.044 (2)	−0.004 (2)	0.0186 (19)	0.0010 (19)
C10	0.031 (2)	0.039 (2)	0.0303 (18)	−0.0002 (17)	0.0116 (15)	0.0001 (16)
C11	0.054 (3)	0.037 (2)	0.048 (2)	−0.0022 (19)	0.027 (2)	−0.0001 (18)
C12	0.056 (3)	0.039 (2)	0.058 (3)	0.004 (2)	0.030 (2)	−0.005 (2)
C13	0.041 (2)	0.063 (3)	0.042 (2)	0.001 (2)	0.0216 (18)	−0.003 (2)
C14	0.095 (4)	0.076 (3)	0.049 (3)	0.012 (3)	0.009 (3)	0.007 (3)

Geometric parameters (Å, º)

Na1—O1	2.294 (3)	C4—C5	1.375 (6)
Na1—O1^{i}	2.294 (3)	C4—H4	0.9300
Na1—O2^{i}	2.344 (2)	C5—C6	1.371 (6)
Na1—O2	2.344 (2)	C5—H5	0.9300
Na1—N2	2.642 (3)	C6—C7	1.382 (5)
Na1—N2^{i}	2.642 (3)	C6—H6	0.9300
Na1—C1^{i}	3.059 (4)	C7—H7	0.9300

N1—C1	1.360 (4)	C8—C10	1.461 (5)
N1—N2	1.390 (4)	C8—H8	0.9300
N1—H1	0.8600	C9—C10	1.391 (5)
N2—C8	1.276 (5)	C9—H9	0.9300
N3—C13	1.330 (5)	C10—C11	1.379 (5)
N3—C9	1.335 (5)	C11—C12	1.371 (5)
O1—C1	1.222 (4)	C11—H11	0.9300
O2—C14	1.412 (5)	C12—C13	1.362 (6)
O2—H2	0.8200	C12—H12	0.9300
C1—C2	1.487 (5)	C13—H13	0.9300
C2—C3	1.377 (5)	C14—H14A	0.9600
C2—C7	1.382 (5)	C14—H14B	0.9600
C3—C4	1.375 (6)	C14—H14C	0.9600
C3—H3	0.9300		

O1—Na1—O1^{i}	180.0	C4—C3—H3	119.8
O1—Na1—O2^{i}	93.26 (10)	C2—C3—H3	119.8
O1^{i}—Na1—O2^{i}	86.74 (10)	C3—C4—C5	121.1 (4)
O1—Na1—O2	86.74 (10)	C3—C4—H4	119.5
O1^{i}—Na1—O2	93.26 (10)	C5—C4—H4	119.5
O2^{i}—Na1—O2	180.0	C6—C5—C4	119.0 (4)
O1—Na1—N2	65.34 (9)	C6—C5—H5	120.5
O1^{i}—Na1—N2	114.66 (9)	C4—C5—H5	120.5
O2^{i}—Na1—N2	91.60 (9)	C5—C6—C7	120.0 (4)
O2—Na1—N2	88.40 (9)	C5—C6—H6	120.0
O1—Na1—N2^{i}	114.66 (9)	C7—C6—H6	120.0
O1^{i}—Na1—N2^{i}	65.34 (9)	C2—C7—C6	121.0 (4)
O2^{i}—Na1—N2^{i}	88.40 (9)	C2—C7—H7	119.5
O2—Na1—N2^{i}	91.60 (9)	C6—C7—H7	119.5
N2—Na1—N2^{i}	180.00 (6)	N2—C8—C10	122.1 (3)
O1—Na1—C1^{i}	159.30 (9)	N2—C8—H8	118.9
O1^{i}—Na1—C1^{i}	20.70 (9)	C10—C8—H8	118.9
O2^{i}—Na1—C1^{i}	76.04 (9)	N3—C9—C10	124.1 (4)
O2—Na1—C1^{i}	103.96 (9)	N3—C9—H9	118.0
N2—Na1—C1^{i}	131.53 (9)	C10—C9—H9	118.0
N2^{i}—Na1—C1^{i}	48.47 (9)	C11—C10—C9	117.5 (3)
C1—N1—N2	119.1 (3)	C11—C10—C8	125.1 (3)
C1—N1—H1	120.4	C9—C10—C8	117.4 (3)
N2—N1—H1	120.4	C12—C11—C10	118.8 (4)
C8—N2—N1	114.5 (3)	C12—C11—H11	120.6
C8—N2—Na1	140.1 (3)	C10—C11—H11	120.6
N1—N2—Na1	102.3 (2)	C13—C12—C11	119.4 (4)
C13—N3—C9	116.3 (3)	C13—C12—H12	120.3
C1—O1—Na1	117.7 (2)	C11—C12—H12	120.3
C14—O2—Na1	122.5 (3)	N3—C13—C12	123.9 (4)
C14—O2—H2	109.5	N3—C13—H13	118.0
Na1—O2—H2	127.7	C12—C13—H13	118.0
O1—C1—N1	121.5 (3)	O2—C14—H14A	109.5

O1—C1—C2	121.5 (3)	O2—C14—H14B	109.5
N1—C1—C2	117.0 (3)	H14A—C14—H14B	109.5
C3—C2—C7	118.4 (3)	O2—C14—H14C	109.5
C3—C2—C1	125.0 (3)	H14A—C14—H14C	109.5
C7—C2—C1	116.6 (3)	H14B—C14—H14C	109.5
C4—C3—C2	120.4 (4)		

C1—N1—N2—C8	−170.8 (3)	N2—N1—C1—C2	178.6 (3)
C1—N1—N2—Na1	25.1 (3)	O1—C1—C2—C3	179.9 (4)
O1—Na1—N2—C8	175.8 (4)	N1—C1—C2—C3	1.0 (5)
O1^{i}—Na1—N2—C8	−4.2 (4)	O1—C1—C2—C7	−0.2 (5)
O2^{i}—Na1—N2—C8	82.9 (4)	N1—C1—C2—C7	−179.1 (3)
O2—Na1—N2—C8	−97.1 (4)	C7—C2—C3—C4	1.1 (6)
C1^{i}—Na1—N2—C8	10.2 (4)	C1—C2—C3—C4	−179.0 (4)
O1—Na1—N2—N1	−27.19 (19)	C2—C3—C4—C5	−0.3 (6)
O1^{i}—Na1—N2—N1	152.81 (19)	C3—C4—C5—C6	−0.6 (7)
O2^{i}—Na1—N2—N1	−120.0 (2)	C4—C5—C6—C7	0.6 (6)
O2—Na1—N2—N1	60.0 (2)	C3—C2—C7—C6	−1.1 (6)
C1^{i}—Na1—N2—N1	167.28 (18)	C1—C2—C7—C6	179.1 (4)
O2^{i}—Na1—O1—C1	122.2 (3)	C5—C6—C7—C2	0.2 (6)
O2—Na1—O1—C1	−57.8 (3)	N1—N2—C8—C10	179.9 (3)
N2—Na1—O1—C1	31.9 (3)	Na1—N2—C8—C10	−24.9 (6)
N2^{i}—Na1—O1—C1	−148.1 (3)	C13—N3—C9—C10	0.9 (6)
C1^{i}—Na1—O1—C1	180.0	N3—C9—C10—C11	−1.0 (6)
O1—Na1—O2—C14	−153.5 (3)	N3—C9—C10—C8	179.3 (4)
O1^{i}—Na1—O2—C14	26.5 (3)	N2—C8—C10—C11	−2.9 (6)
N2—Na1—O2—C14	141.2 (3)	N2—C8—C10—C9	176.7 (4)
N2^{i}—Na1—O2—C14	−38.8 (3)	C9—C10—C11—C12	0.5 (5)
C1^{i}—Na1—O2—C14	8.6 (4)	C8—C10—C11—C12	−179.9 (4)
Na1—O1—C1—N1	−32.3 (4)	C10—C11—C12—C13	0.0 (6)
Na1—O1—C1—C2	148.9 (3)	C9—N3—C13—C12	−0.3 (6)
N2—N1—C1—O1	−0.3 (5)	C11—C12—C13—N3	−0.1 (7)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···I1^{ii}	0.86	3.03	3.816 (3)	153
O2—H2···N3^{iii}	0.82	1.98	2.792 (5)	171

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