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2,2'-(Piperazine-1,4-diyl)diethanaminium bis(2-hydroxybenzoate)

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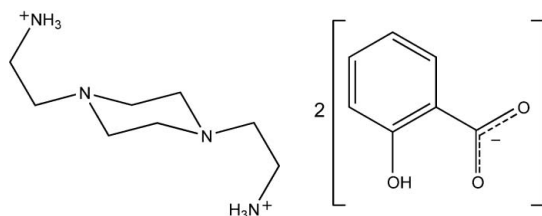
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Key indicators: single-crystal X-ray study; $T = 180$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.050; wR factor = 0.147; data-to-parameter ratio = 20.8.

The asymmetric unit of the title salt, $\text{C}_8\text{H}_{22}\text{N}_4^{2+} \cdot 2\text{C}_7\text{H}_5\text{O}_3^-$, comprises half a 2,2'-(piperazine-1,4-diyl)diethanaminium dication plus a 2-hydroxybenzoate anion. In the crystal, the anions and cations are linked by $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds to form infinite two-dimensional networks parallel with the a unit-cell face. The conformation adopted by the cation in the crystal is very similar to that adopted by the same cation in the structures of the nitrate and tetrahydrogen pentaborate salts.

Related literature

For the structures of the nitrate and tetrahydrogen pentaborate salts of the 1,4-di(2-ammonioethyl)piperazine cation, see: Junk & Smith (2005); Jiang *et al.* (2009); Yang *et al.* (2011).



Experimental

Crystal data

 $\text{C}_8\text{H}_{22}\text{N}_4^{2+} \cdot 2\text{C}_7\text{H}_5\text{O}_3^-$ $M_r = 448.52$ Monoclinic, $P2_1/c$ $a = 11.5374$ (4) Å $b = 6.4759$ (2) Å $c = 15.5264$ (6) Å $\beta = 104.207$ (2)° $V = 1124.58$ (7) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.10$ mm⁻¹ $T = 180$ K $0.37 \times 0.10 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer

Absorption correction: multi-scan

(SORTAV; Blessing, 1995)

 $T_{\min} = 0.852$, $T_{\max} = 0.995$

19655 measured reflections

3261 independent reflections

2013 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.065$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$ $wR(F^2) = 0.147$ $S = 1.08$

3261 reflections

157 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1A} \cdots \text{O14}$	0.929 (17)	1.992 (18)	2.8853 (17)	160.9 (14)
$\text{N1}-\text{H1B} \cdots \text{O14}^i$	0.900 (18)	1.923 (19)	2.7909 (17)	161.5 (14)
$\text{N1}-\text{H1C} \cdots \text{O15}^{ii}$	0.892 (18)	1.902 (19)	2.7843 (17)	169.9 (15)
$\text{O16}-\text{H16} \cdots \text{O15}$	0.81 (2)	1.83 (2)	2.5641 (16)	149.4 (19)

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

Data collection: COLLECT (Nonius, 1998); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO (Otwinowski & Minor, 1997), SCALEPACK and SORTAV (Blessing, 1995); program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997), POV-RAY (Cason, 2004) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2133).

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supporting information

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2,2'-(Piperazine-1,4-diyl)diethanaminium bis(2-hydroxybenzoate)**Ignacy Cukrowski, Adedapo S. Adeyinka and David C. Liles****S1. Comment**

The title compound [$C_8H_{22}N_4^{2+} 2(C_7H_5O_3^-)$] (**1**) was obtained as an unintended product during an attempt to prepare a 2-hydroxybenzoate salt of a singly protonated *N,N'*-di(2-aminoethyl)-2-aminoethane-1-ammonium ion ($C_6H_{19}N_4^+ C_7H_5O_3^-$). This occurred because the starting material, instead of being pure *N,N'*-di(2-aminoethyl)-ethane-1,2-diamine ($C_6H_{18}N_4$), was a mixture of that compound and 1,4-di(2-aminoethyl)piperazine ($C_8H_{22}N_4$). A similar situation appears to have occurred for a published structure which the authors (Yang, *et al.*, 2011) claim to be a *N,N'*-di(2-ammonioethyl)-ethane-1,2-diamine (*i.e.* a doubly protonated ion derived from $C_6H_{18}N_4$) salt of tetrahydrogenpentaborate ($H_4B_5O_{10}^-$) but with the central C_2H_4 moiety disordered over two sites. In fact the two "disordered" C_2H_4 sites together with the two adjacent N atoms form the central piperazine ring of a 1,4-di(2-ammonioethyl)piperazine ion ($C_8H_{22}N_4^{2+}$) and the reported crystal structure is identical (within experimental error) with that of $C_8H_{22}N_4^{2+} 2(H_4B_5O_{10}^-)$ (Jiang, *et al.*, 2009).

The $C_8H_{22}N_4^{2+}$ cation in **1** is symmetrical and lies across a crystallographic centre of inversion. Each ammonium group in the cations of **1** is the donor for three hydrogen bonds to the O atoms of the carboxylate groups of three different 2-hydroxybenzoate anions (Fig. 1). There is also an intra-molecular hydrogen bond between the hydroxy group and one of the carboxylate O atoms in the $C_7H_5O_3^-$ anions. Thus both the O atoms of each carboxylate group are each acceptors for two hydrogen bonds. The hydrogen bonds link the cations and anions to form two-dimensional networks with the layers parallel with the *A* face of the unit cell (Fig. 2).

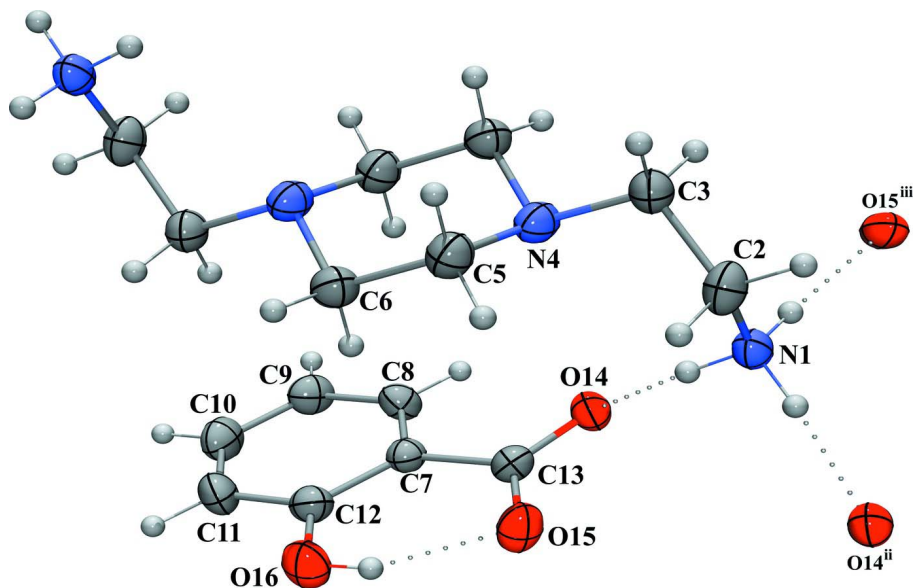
Fig. 3 illustrates that the conformation adopted by the $C_8H_{22}N_4^{2+}$ cation in the crystal structure of **1** is very similar to the conformations adopted by the same cation in the crystal structures of the NO_3^- (Junk & Smith, 2005) and the $H_4B_5O_{10}^-$ (Jiang, *et al.*, 2009) salts despite the differences in the size and shape of the anions in the various structures.

S2. Experimental

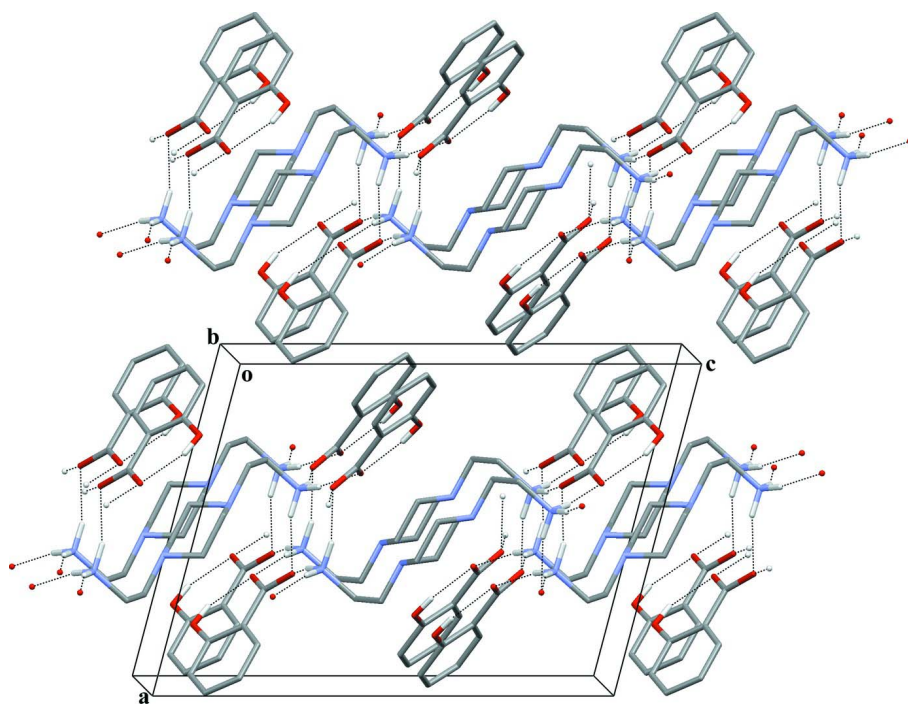
2 ml of a 3.32 *M* aqueous solution of what was claimed by the supplier (QinHuangDao JinLei Chemical Co.Ltd) to be *N,N'*-di(2-aminoethyl)-ethane-1,2-diamine, but which turned out to be a mixture of that compound ($C_6H_{18}N_4$, 6.64 *mmol*) and 1,4-di(2-aminoethyl)piperazine ($C_8H_{20}N_4$, 5.57(1-*n*) *mmol*) was added to 0.96 g of 2-hydroxybenzoic acid (6.95 *mmol*), resulting in a clear colourless solution. 0.2 ml of ethanol was added to the solution and the mixture was heated for 3 h at 70 °C, the solution turned greenish yellow after one hour of heating. It was cooled to room temperature and then left covered for six days and then allowed to slowly evaporate by covering the container with perforated aluminium foil. Yellow crystals were obtained after four days of slow evaporation.

S3. Refinement

H1A, H1B and H1C were located by a difference map and their coordinates were refined. All of the remaining H atoms were placed in their calculated positions and then refined using the riding model with Atom—H lengths of 0.95 Å, (CH) or 0.99 Å (CH₂). Isotropic displacement parameters for all hydrogen atoms were set to 1.20 times U_{eq} of the parent atom.

**Figure 1**

Molecular structure of the title compound showing the atom labeling scheme and 50° probability displacement ellipsoids. Broken lines indicate N—H···O and O—H···O hydrogen bonds. Symmetry codes: (ii) $-x + 1, y - 1/2, -z + 3/2$; (iii) $-x + 1, y + 1/2, -z + 3/2$.

**Figure 2**

Packing diagram of the title compound viewed offset from along the *b* axis. Dashed lines indicate N—H···O and O—H···O hydrogen bonds. The intermolecular N—H···O hydrogen bonds form a two-dimensional network.

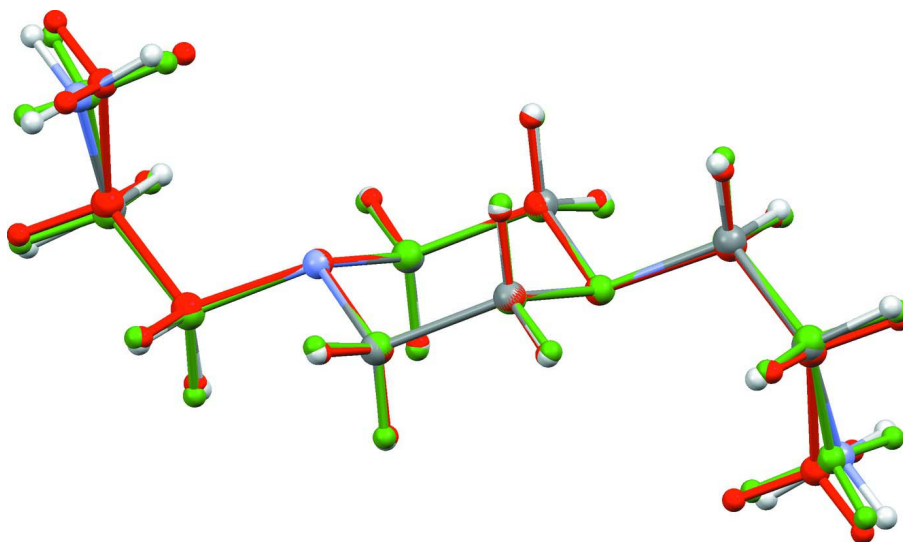


Figure 3

The structures of the 1,4-di(2-ammonioethyl)piperazine cations of the $C_7H_5O_3^-$ (standard atom colours, **1**), the NO_3^- (green, Junk & Smith, 2005) and the $H_4B_5O_{10}^-$ (red, Jiang, *et al.*, 2009) salts, superimposed on each other to show the similar conformations adopted by the cations in all three structures.

2,2'-(Piperazine-1,4-diyl)diethanaminium bis(2-hydroxybenzoate)

Crystal data

$C_8H_{22}N_4^{2+} \cdot 2C_7H_5O_3^-$

$M_r = 448.52$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.5374$ (4) Å

$b = 6.4759$ (2) Å

$c = 15.5264$ (6) Å

$\beta = 104.207$ (2)°

$V = 1124.58$ (7) Å³

$Z = 2$

$F(000) = 480$

$D_x = 1.325$ Mg m⁻³

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

Cell parameters from 26933 reflections

$\theta = 1.0\text{--}30.0^\circ$

$\mu = 0.10$ mm⁻¹

$T = 180$ K

Block, yellow

$0.37 \times 0.10 \times 0.05$ mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Thin slice ω and φ scans

Absorption correction: multi-scan
(*SORTAV*; Blessing, 1995)

$T_{\min} = 0.852$, $T_{\max} = 0.995$

19655 measured reflections

3261 independent reflections

2013 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.065$

$\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.6^\circ$

$h = -16 \rightarrow 16$

$k = -8 \rightarrow 9$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.147$

$S = 1.08$

3261 reflections

157 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 0.0149P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.34 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.28 \text{ e } \text{Å}^{-3}$

Special details

Experimental. The –OH and –NH₃ hydrogen atoms were located and their positions were refined satisfactorily.

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.39360 (12)	0.1995 (2)	0.71495 (9)	0.0296 (3)
H1A	0.4724 (16)	0.215 (2)	0.7117 (11)	0.036*
H1B	0.3890 (14)	0.076 (3)	0.7403 (10)	0.036*
H1C	0.3822 (14)	0.299 (3)	0.7516 (11)	0.036*
C2	0.30579 (14)	0.2076 (2)	0.62703 (10)	0.0355 (4)
H2A	0.2242	0.1866	0.6352	0.043*
H2B	0.3228	0.0945	0.5890	0.043*
C3	0.31138 (13)	0.4123 (2)	0.58149 (11)	0.0328 (4)
H3A	0.2467	0.4186	0.5262	0.039*
H3B	0.2985	0.5257	0.6209	0.039*
N4	0.42749 (11)	0.44057 (19)	0.55982 (8)	0.0321 (3)
C5	0.43393 (14)	0.3166 (2)	0.48157 (11)	0.0350 (4)
H5A	0.3691	0.3593	0.4302	0.042*
H5B	0.4219	0.1691	0.4937	0.042*
C6	0.44661 (14)	0.6567 (2)	0.54111 (10)	0.0332 (4)
H6A	0.4446	0.7419	0.5937	0.040*
H6B	0.3817	0.7044	0.4907	0.040*
C7	0.80219 (12)	0.2749 (2)	0.66915 (9)	0.0261 (3)
C8	0.84808 (13)	0.4670 (2)	0.70044 (10)	0.0307 (3)
H8	0.8129	0.5394	0.7408	0.037*
C9	0.94297 (13)	0.5555 (2)	0.67466 (11)	0.0356 (4)
H9	0.9731	0.6863	0.6973	0.043*
C10	0.99343 (14)	0.4510 (3)	0.61536 (11)	0.0390 (4)
H10	1.0584	0.5111	0.5968	0.047*
C11	0.95065 (15)	0.2608 (3)	0.58287 (11)	0.0381 (4)
H11	0.9861	0.1908	0.5420	0.046*
C12	0.85550 (13)	0.1702 (2)	0.60957 (10)	0.0306 (3)
C13	0.69936 (13)	0.1830 (2)	0.69894 (9)	0.0273 (3)
O14	0.64524 (9)	0.28943 (15)	0.74448 (7)	0.0321 (3)

O15	0.67079 (11)	-0.00229 (16)	0.67621 (7)	0.0393 (3)
O16	0.81677 (11)	-0.01682 (18)	0.57563 (8)	0.0418 (3)
H16	0.7652 (17)	-0.055 (3)	0.5996 (14)	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0299 (7)	0.0264 (7)	0.0360 (7)	-0.0006 (6)	0.0145 (6)	0.0013 (6)
C2	0.0369 (8)	0.0391 (9)	0.0326 (8)	-0.0098 (7)	0.0124 (7)	-0.0004 (7)
C3	0.0312 (8)	0.0365 (9)	0.0327 (8)	0.0006 (7)	0.0115 (6)	0.0030 (7)
N4	0.0301 (7)	0.0310 (7)	0.0359 (7)	-0.0020 (5)	0.0091 (5)	0.0064 (6)
C5	0.0381 (8)	0.0336 (8)	0.0345 (9)	-0.0029 (7)	0.0111 (7)	0.0020 (7)
C6	0.0362 (8)	0.0299 (8)	0.0339 (8)	0.0013 (7)	0.0094 (7)	0.0017 (7)
C7	0.0249 (7)	0.0260 (7)	0.0270 (7)	0.0011 (6)	0.0057 (6)	0.0028 (6)
C8	0.0265 (7)	0.0302 (8)	0.0359 (8)	0.0007 (6)	0.0084 (6)	-0.0030 (7)
C9	0.0298 (8)	0.0322 (8)	0.0435 (9)	-0.0052 (7)	0.0068 (7)	0.0011 (7)
C10	0.0289 (8)	0.0464 (10)	0.0434 (9)	-0.0028 (7)	0.0123 (7)	0.0066 (8)
C11	0.0355 (8)	0.0446 (10)	0.0387 (9)	0.0066 (7)	0.0180 (7)	0.0010 (7)
C12	0.0332 (8)	0.0278 (7)	0.0309 (8)	0.0021 (6)	0.0076 (6)	0.0013 (6)
C13	0.0294 (7)	0.0254 (7)	0.0266 (7)	-0.0011 (6)	0.0061 (6)	0.0025 (6)
O14	0.0321 (6)	0.0299 (6)	0.0379 (6)	0.0002 (4)	0.0154 (5)	-0.0010 (5)
O15	0.0528 (7)	0.0291 (6)	0.0409 (6)	-0.0125 (5)	0.0210 (5)	-0.0062 (5)
O16	0.0532 (8)	0.0314 (6)	0.0466 (7)	-0.0046 (5)	0.0234 (6)	-0.0111 (5)

Geometric parameters (Å, °)

N1—C2	1.488 (2)	C6—H6B	0.9900
N1—H1A	0.929 (17)	C7—C8	1.392 (2)
N1—H1B	0.900 (18)	C7—C12	1.404 (2)
N1—H1C	0.892 (18)	C7—C13	1.498 (2)
C2—C3	1.511 (2)	C8—C9	1.379 (2)
C2—H2A	0.9900	C8—H8	0.9500
C2—H2B	0.9900	C9—C10	1.381 (2)
C3—N4	1.4706 (19)	C9—H9	0.9500
C3—H3A	0.9900	C10—C11	1.376 (2)
C3—H3B	0.9900	C10—H10	0.9500
N4—C6	1.4572 (19)	C11—C12	1.394 (2)
N4—C5	1.473 (2)	C11—H11	0.9500
C5—C6 ⁱ	1.514 (2)	C12—O16	1.3524 (18)
C5—H5A	0.9900	C13—O14	1.2575 (17)
C5—H5B	0.9900	C13—O15	1.2714 (17)
C6—C5 ⁱ	1.514 (2)	O16—H16	0.81 (2)
C6—H6A	0.9900		
C2—N1—H1A	113.7 (10)	N4—C6—H6A	109.6
C2—N1—H1B	109.4 (10)	C5 ⁱ —C6—H6A	109.6
H1A—N1—H1B	106.6 (14)	N4—C6—H6B	109.6
C2—N1—H1C	112.7 (10)	C5 ⁱ —C6—H6B	109.6

H1A—N1—H1C	104.7 (15)	H6A—C6—H6B	108.1
H1B—N1—H1C	109.4 (15)	C8—C7—C12	118.06 (14)
N1—C2—C3	111.24 (13)	C8—C7—C13	120.61 (13)
N1—C2—H2A	109.4	C12—C7—C13	121.33 (13)
C3—C2—H2A	109.4	C9—C8—C7	122.09 (14)
N1—C2—H2B	109.4	C9—C8—H8	119.0
C3—C2—H2B	109.4	C7—C8—H8	119.0
H2A—C2—H2B	108.0	C8—C9—C10	118.95 (15)
N4—C3—C2	111.24 (12)	C8—C9—H9	120.5
N4—C3—H3A	109.4	C10—C9—H9	120.5
C2—C3—H3A	109.4	C11—C10—C9	120.74 (15)
N4—C3—H3B	109.4	C11—C10—H10	119.6
C2—C3—H3B	109.4	C9—C10—H10	119.6
H3A—C3—H3B	108.0	C10—C11—C12	120.36 (15)
C6—N4—C3	110.87 (12)	C10—C11—H11	119.8
C6—N4—C5	108.55 (12)	C12—C11—H11	119.8
C3—N4—C5	110.73 (11)	O16—C12—C11	118.04 (14)
N4—C5—C6 ⁱ	111.26 (12)	O16—C12—C7	122.17 (13)
N4—C5—H5A	109.4	C11—C12—C7	119.79 (14)
C6 ⁱ —C5—H5A	109.4	O14—C13—O15	122.86 (13)
N4—C5—H5B	109.4	O14—C13—C7	119.76 (13)
C6 ⁱ —C5—H5B	109.4	O15—C13—C7	117.39 (13)
H5A—C5—H5B	108.0	C12—O16—H16	107.4 (14)
N4—C6—C5 ⁱ	110.31 (13)		
N1—C2—C3—N4	64.77 (17)	C9—C10—C11—C12	-0.1 (2)
C2—C3—N4—C6	-162.96 (13)	C10—C11—C12—O16	179.96 (14)
C2—C3—N4—C5	76.48 (16)	C10—C11—C12—C7	0.7 (2)
C6—N4—C5—C6 ⁱ	58.44 (17)	C8—C7—C12—O16	-179.88 (13)
C3—N4—C5—C6 ⁱ	-179.64 (12)	C13—C7—C12—O16	0.5 (2)
C3—N4—C6—C5 ⁱ	-179.69 (12)	C8—C7—C12—C11	-0.7 (2)
C5—N4—C6—C5 ⁱ	-57.85 (17)	C13—C7—C12—C11	179.64 (13)
C12—C7—C8—C9	0.1 (2)	C8—C7—C13—O14	8.1 (2)
C13—C7—C8—C9	179.72 (13)	C12—C7—C13—O14	-172.27 (13)
C7—C8—C9—C10	0.5 (2)	C8—C7—C13—O15	-172.00 (13)
C8—C9—C10—C11	-0.5 (2)	C12—C7—C13—O15	7.6 (2)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O14	0.929 (17)	1.992 (18)	2.8853 (17)	160.9 (14)
N1—H1B \cdots O14 ⁱⁱ	0.900 (18)	1.923 (19)	2.7909 (17)	161.5 (14)
N1—H1C \cdots O15 ⁱⁱⁱ	0.892 (18)	1.902 (19)	2.7843 (17)	169.9 (15)
O16—H16 \cdots O15	0.81 (2)	1.83 (2)	2.5641 (16)	149.4 (19)

Symmetry codes: (ii) $-x+1, y-1/2, -z+3/2$; (iii) $-x+1, y+1/2, -z+3/2$.