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2-Amino-4-methylpyridinium 6-carboxypyridine-2-carboxylate methanol monosolvate

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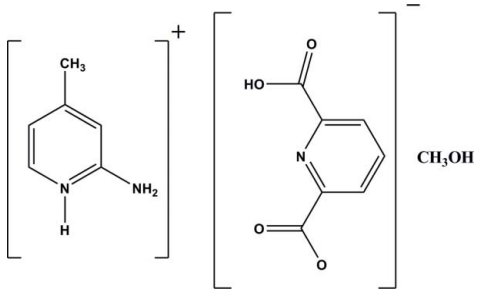
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.079; wR factor = 0.216; data-to-parameter ratio = 18.0.

In the title solvated molecular salt, $C_6H_9N_2^+\cdot C_7H_4NO_4^-\cdot CH_4O$, the pyridine N atom of 2-amino-4-methylpyridine is protonated and one carboxyl group of pyridine-2,6-dicarboxylic acid is deprotonated. The dihedral angles between the $-CO_2$ and –COH groups and the pyridine ring are 0.65 (13) and 7.4°. The crystal packing is stabilized by intermolecular N—H···O, O—H···O and weak C—H···O hydrogen bonds.

Related literature

For background to proton-transfer compounds, see: Aghabozorg *et al.* (2008). For related structures, see: Aakeröy *et al.* (1998); Aghabozorg *et al.* (2006); Al-Allaf *et al.* (2003); Fu *et al.* (2005); Linden *et al.* (2003); Moghimi *et al.* (2004); Sheshmani *et al.* (2006); Thanigaimani *et al.* (2007).



Experimental

Crystal data

$C_6H_9N_2^+\cdot C_7H_4NO_4^-\cdot CH_4O$
M_r = 307.31
Triclinic, $P\overline{1}$
a = 7.2191 (14) Å
b = 9.5095 (19) Å
c = 11.139 (2) Å
α = 94.44 (3)°
β = 99.76 (3)°
γ = 92.50 (3)°
V = 750.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.4 × 0.25 × 0.2 mm

Data collection

Stoe IPDS II diffractometer
8658 measured reflections
4005 independent reflections
2697 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.216
S = 1.17
4005 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14C···O2^{i}	0.96	2.59	3.488 (5)	157
O5—H5A···O3^{ii}	0.78 (5)	2.02 (5)	2.796 (3)	170 (4)
N3—H3B···O4^{iii}	0.83 (4)	1.95 (4)	2.764 (3)	166 (3)
N3—H3A···O2^{i}	0.87 (4)	2.30 (4)	3.122 (3)	158 (3)
N2—H2···O3^{iii}	0.85 (3)	1.87 (3)	2.723 (3)	173 (3)
O1—H1···O5^{iv}	0.87 (4)	1.87 (4)	2.689 (3)	156 (4)

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5416).

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2-Amino-4-methylpyridinium 6-carboxypyridine-2-carboxylate methanol monosolvate

Hossein Aghabozorg, Azadeh Mofidi Rouchi, Masoud Mirzaei and Behrouz Notash

S1. Comment

Continuing the path to synthesize proton transfer compounds, our group have been focused on forming ion pairs between 2,6-pydcH_2 and various organic bases (Aghabozorg *et al.*, 2008). Due to its flat and symmetric structure and two proton donor sites, 2,6-pydcH_2 has a potential of constructing supramolecular networks. Proton transfer compounds of 2,6-pydcH_2 with nitrogen donor molecules such as 2-chloro-benzylamine (Aakeröy *et al.*,1998), piperazine (Aghabozorg *et al.*, 2006 & Sheshmani *et al.*, 2006), phenanthroline (Fu *et al.*, 2005), creatinine (Moghimi *et al.*, 2004) and 2-amino-4,6-dimethoxypyrimidine (Thanigaimani *et al.*, 2007) have been synthesized and characterized by single-crystal X-ray diffraction method. In addition, the formation of monoprotonated 2-amino-4-methylpyridine (2a4mpH) has been reported in several proton transfer systems (Al-Allaf *et al.*, 2003; Linden *et al.* 2003).

The title compound, (2a4mpH)(2,6-pydcH).CH_3OH, consist of one mono deprotonated 2,6-pydcH_2 unit, one mono protonated 2a4mp, and one methanol molecule. The asymmetric unit of the title compound is shown in Fig. 1. The title compound, was formed from the reaction between 2,6-pydcH_2 as a proton donor and 2a4mp as a proton acceptor. There are several N—H···O, O—H···O and weak C—H···O hydrogen bonds, in crystal structure of the title compound (Table 1 & Fig. 2). The crystal structure shows that one of the protons of carboxylic groups has been transferred to $N_{pyridine}$ of 2a4mp. Indeed, the structure formed self-assembled supramolecular network through noncovalent interactions.

S2. Experimental

The reaction between a solution of 2,6-pydcH_2 (0.1671 mg, 1 mmol) in 10 ml water and 2a4mp (0.2163 mg, 2 mmol) in 10 ml methanol in 1:2 molar ratios gave block colorless crystals of the title compound after slow evaporation of the solvent at room temperature (m.p: 267).

S3. Refinement

The hydrogen atoms bonded to N and O were found in a difference Fourier map and refined isotropically. The C—H protons were positioned geometrically and refined as riding atoms with C—H = 0.93 Å and *U*iso(H) = 1.2 *U*eq(C) for aromatic C—H groups and C—H = 0.98 Å and *U*iso(H) = 1.5 *Ueq*(C) for methyl group.

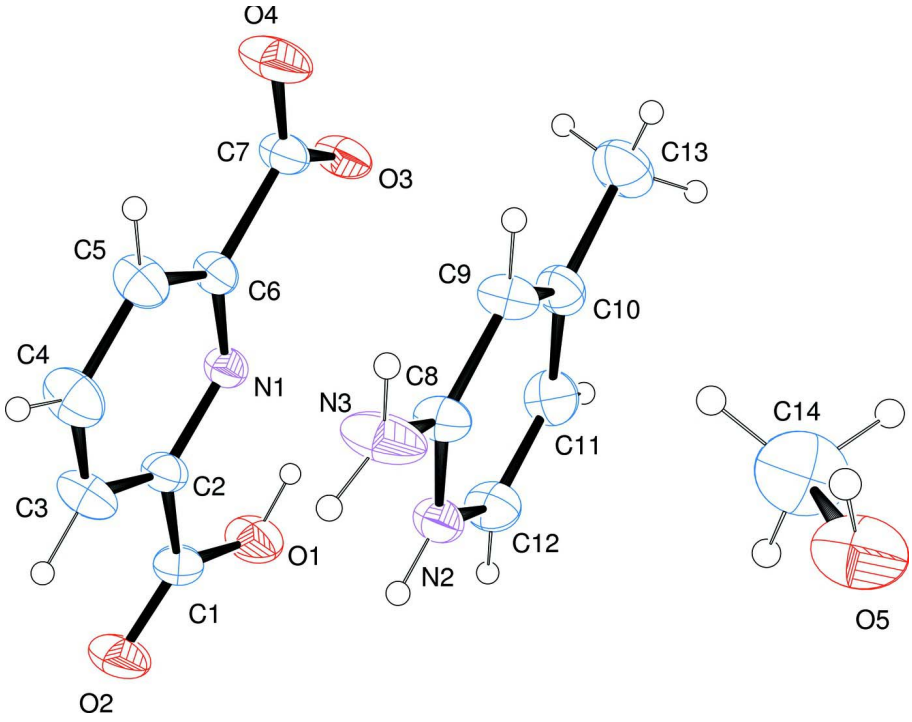

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at 30% probability level.

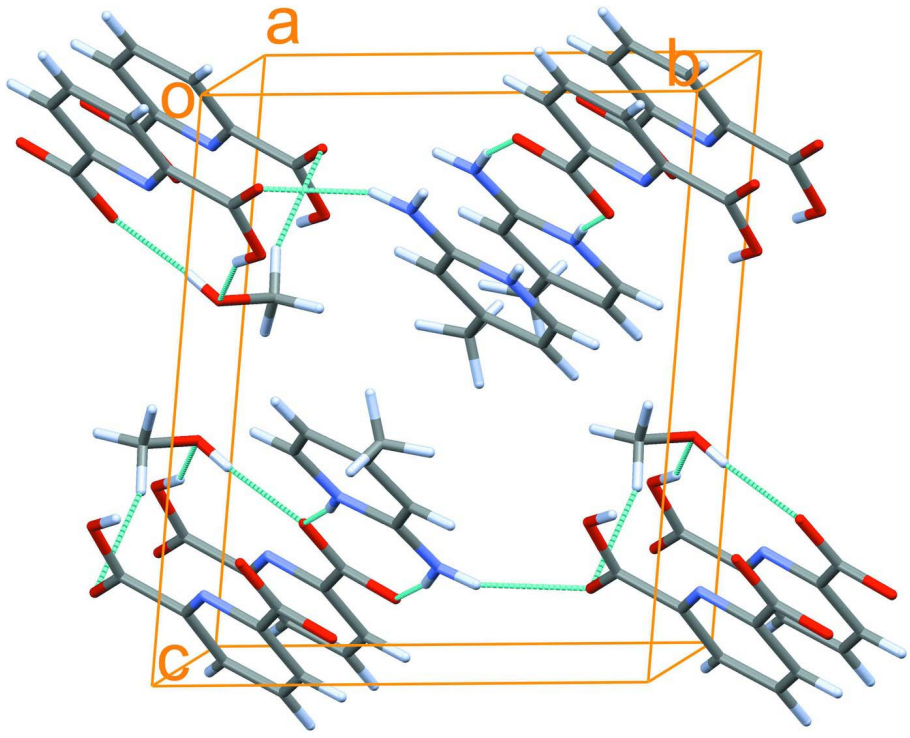

Figure 2

The packing diagram of the title compound. The intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds are shown as blue dashed lines.

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Crystal data

$C_6H_9N_2^+·C_7H_4NO_4^-·CH_4O$
$M_r = 307.31$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.2191$ (14) Å
$b = 9.5095$ (19) Å
$c = 11.139$ (2) Å
$\alpha = 94.44$ (3)°
$\beta = 99.76$ (3)°
$\gamma = 92.50$ (3)°
$V = 750.1$ (3) Å^3
$Z = 2$
$F(000) = 324.0$
$D_x = 1.361$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4005 reflections
$\theta = 2.2$–$29.2°$
$\mu = 0.11$ mm^{-1}
$T = 298$ K
Block, colorless
0.4 × 0.25 × 0.2 mm

Data collection

Stoe IPDS II diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.15 mm pixels mm^{-1}
rotation method scans
8658 measured reflections
4005 independent reflections
2697 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 29.2°$, $\theta_{min} = 2.2°$
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 13$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.079$
$wR(F^2) = 0.216$
$S = 1.17$
4005 reflections
222 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0939P)^2 + 0.144P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.07 (2)

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}
O1	0.0312 (3)	−0.1514 (2)	0.73074 (19)	0.0679 (6)
O2	0.2937 (3)	−0.1669 (2)	0.8602 (2)	0.0686 (6)
O3	−0.3748 (2)	0.20309 (19)	0.76854 (17)	0.0544 (5)

O4	−0.2965 (3)	0.3911 (2)	0.9022 (2)	0.0730 (7)
O5	0.7131 (3)	0.9637 (2)	0.6320 (2)	0.0685 (6)
N1	−0.0374 (3)	0.09454 (18)	0.84467 (16)	0.0381 (4)
N2	0.2829 (3)	0.3098 (2)	0.70000 (18)	0.0424 (5)
N3	0.3671 (4)	0.5125 (3)	0.8268 (3)	0.0689 (8)
C2	0.1276 (3)	0.0410 (2)	0.8845 (2)	0.0415 (5)
C3	0.2618 (4)	0.1087 (3)	0.9759 (3)	0.0567 (7)
H3	0.3748	0.0678	1.0017	0.068*
C4	0.2249 (4)	0.2381 (3)	1.0281 (3)	0.0587 (7)
H4	0.3135	0.2872	1.0890	0.070*
C5	0.0548 (4)	0.2936 (2)	0.9886 (2)	0.0501 (6)
H5	0.0264	0.3806	1.0229	0.060*
C6	−0.0741 (3)	0.2185 (2)	0.8971 (2)	0.0386 (5)
C1	0.1592 (4)	−0.1007 (3)	0.8243 (2)	0.0495 (6)
C7	−0.2648 (3)	0.2756 (2)	0.8524 (2)	0.0455 (5)
C8	0.2394 (3)	0.4369 (2)	0.7460 (2)	0.0447 (5)
C9	0.0583 (3)	0.4838 (3)	0.7033 (2)	0.0509 (6)
H9	0.0248	0.5712	0.7337	0.061*
C10	−0.0679 (3)	0.4020 (3)	0.6179 (2)	0.0478 (6)
C11	−0.0140 (4)	0.2706 (3)	0.5725 (2)	0.0545 (6)
H11	−0.0972	0.2136	0.5138	0.065*
C12	0.1599 (4)	0.2277 (3)	0.6147 (2)	0.0515 (6)
H12	0.1953	0.1408	0.5848	0.062*
C13	−0.2608 (4)	0.4518 (4)	0.5746 (3)	0.0691 (8)
H13A	−0.3498	0.4075	0.6168	0.104*
H13B	−0.2964	0.4272	0.4882	0.104*
H13C	−0.2592	0.5525	0.5910	0.104*
C14	0.5526 (5)	0.8734 (4)	0.6264 (4)	0.0877 (11)
H14A	0.5833	0.7775	0.6097	0.132*
H14B	0.4556	0.8979	0.5625	0.132*
H14C	0.5088	0.8831	0.7032	0.132*
H5A	0.700 (6)	1.030 (5)	0.676 (4)	0.097 (14)*
H2	0.392 (5)	0.282 (3)	0.727 (3)	0.058 (8)*
H1	−0.058 (6)	−0.094 (4)	0.713 (4)	0.095 (12)*
H3A	0.338 (5)	0.595 (4)	0.855 (3)	0.084 (11)*
H3B	0.465 (5)	0.479 (4)	0.861 (3)	0.068 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0737 (14)	0.0578 (11)	0.0673 (13)	0.0256 (10)	0.0023 (10)	−0.0158 (9)
O2	0.0602 (12)	0.0590 (11)	0.0874 (14)	0.0301 (9)	0.0100 (10)	0.0034 (10)
O3	0.0426 (9)	0.0509 (9)	0.0627 (11)	0.0152 (7)	−0.0078 (8)	−0.0096 (8)
O4	0.0609 (12)	0.0584 (11)	0.0882 (15)	0.0281 (9)	−0.0126 (10)	−0.0246 (10)
O5	0.0592 (12)	0.0678 (13)	0.0696 (13)	0.0078 (10)	−0.0027 (10)	−0.0227 (10)
N1	0.0377 (9)	0.0360 (9)	0.0402 (9)	0.0065 (7)	0.0046 (7)	0.0020 (7)
N2	0.0400 (10)	0.0427 (10)	0.0432 (10)	0.0103 (8)	0.0031 (8)	0.0000 (8)
N3	0.0466 (13)	0.0550 (13)	0.0925 (19)	0.0163 (11)	−0.0104 (12)	−0.0305 (13)

C2	0.0390 (11)	0.0390 (10)	0.0482 (12)	0.0102 (8)	0.0089 (9)	0.0069 (9)
C3	0.0380 (12)	0.0537 (14)	0.0738 (17)	0.0107 (10)	−0.0055 (11)	0.0060 (12)
C4	0.0488 (14)	0.0515 (14)	0.0654 (16)	−0.0006 (11)	−0.0137 (12)	−0.0053 (12)
C5	0.0527 (14)	0.0368 (11)	0.0546 (14)	0.0052 (10)	−0.0049 (11)	−0.0040 (10)
C6	0.0381 (11)	0.0355 (10)	0.0408 (11)	0.0061 (8)	0.0020 (8)	0.0031 (8)
C1	0.0512 (14)	0.0451 (12)	0.0546 (14)	0.0161 (10)	0.0137 (11)	0.0014 (10)
C7	0.0418 (12)	0.0420 (11)	0.0506 (13)	0.0132 (9)	0.0021 (9)	−0.0014 (9)
C8	0.0387 (11)	0.0413 (11)	0.0526 (13)	0.0072 (9)	0.0058 (9)	−0.0029 (9)
C9	0.0421 (12)	0.0462 (12)	0.0648 (15)	0.0127 (10)	0.0091 (11)	0.0011 (11)
C10	0.0370 (11)	0.0550 (13)	0.0519 (13)	0.0036 (10)	0.0043 (10)	0.0136 (10)
C11	0.0503 (14)	0.0585 (15)	0.0491 (14)	−0.0037 (11)	−0.0016 (11)	−0.0042 (11)
C12	0.0538 (14)	0.0449 (12)	0.0530 (14)	0.0051 (10)	0.0067 (11)	−0.0077 (10)
C13	0.0419 (14)	0.081 (2)	0.083 (2)	0.0088 (13)	−0.0001 (13)	0.0215 (16)
C14	0.078 (2)	0.076 (2)	0.106 (3)	−0.0007 (18)	0.012 (2)	0.003 (2)

Geometric parameters (Å, º)

O1—C1	1.315 (3)	C4—C5	1.375 (4)
O1—H1	0.87 (4)	C4—H4	0.9300
O2—C1	1.208 (3)	C5—C6	1.386 (3)
O3—C7	1.257 (3)	C5—H5	0.9300
O4—C7	1.240 (3)	C6—C7	1.520 (3)
O5—C14	1.401 (4)	C8—C9	1.417 (3)
O5—H5A	0.78 (5)	C9—C10	1.367 (4)
N1—C6	1.332 (3)	C9—H9	0.9300
N1—C2	1.335 (3)	C10—C11	1.407 (4)
N2—C8	1.347 (3)	C10—C13	1.504 (4)
N2—C12	1.356 (3)	C11—C12	1.356 (4)
N2—H2	0.85 (3)	C11—H11	0.9300
N3—C8	1.318 (3)	C12—H12	0.9300
N3—H3A	0.87 (4)	C13—H13A	0.9600
N3—H3B	0.83 (4)	C13—H13B	0.9600
C2—C3	1.378 (4)	C13—H13C	0.9600
C2—C1	1.503 (3)	C14—H14A	0.9600
C3—C4	1.376 (4)	C14—H14B	0.9600
C3—H3	0.9300	C14—H14C	0.9600
C1—O1—H1	112 (3)	O3—C7—C6	117.74 (19)
C14—O5—H5A	106 (3)	N3—C8—N2	118.9 (2)
C6—N1—C2	118.17 (19)	N3—C8—C9	123.1 (2)
C8—N2—C12	122.1 (2)	N2—C8—C9	118.0 (2)
C8—N2—H2	118 (2)	C10—C9—C8	120.7 (2)
C12—N2—H2	120 (2)	C10—C9—H9	119.6
C8—N3—H3A	118 (2)	C8—C9—H9	119.6
C8—N3—H3B	123 (2)	C9—C10—C11	118.7 (2)
H3A—N3—H3B	118 (3)	C9—C10—C13	120.3 (3)
N1—C2—C3	123.2 (2)	C11—C10—C13	121.0 (3)
N1—C2—C1	115.8 (2)	C12—C11—C10	119.6 (2)

C3—C2—C1	121.0 (2)	C12—C11—H11	120.2
C4—C3—C2	118.5 (2)	C10—C11—H11	120.2
C4—C3—H3	120.8	C11—C12—N2	120.9 (2)
C2—C3—H3	120.8	C11—C12—H12	119.5
C5—C4—C3	118.9 (2)	N2—C12—H12	119.5
C5—C4—H4	120.6	C10—C13—H13A	109.5
C3—C4—H4	120.6	C10—C13—H13B	109.5
C4—C5—C6	119.3 (2)	H13A—C13—H13B	109.5
C4—C5—H5	120.3	C10—C13—H13C	109.5
C6—C5—H5	120.3	H13A—C13—H13C	109.5
N1—C6—C5	122.0 (2)	H13B—C13—H13C	109.5
N1—C6—C7	117.22 (19)	O5—C14—H14A	109.5
C5—C6—C7	120.77 (19)	O5—C14—H14B	109.5
O2—C1—O1	121.0 (2)	H14A—C14—H14B	109.5
O2—C1—C2	122.2 (2)	O5—C14—H14C	109.5
O1—C1—C2	116.8 (2)	H14A—C14—H14C	109.5
O4—C7—O3	125.9 (2)	H14B—C14—H14C	109.5
O4—C7—C6	116.3 (2)		

C6—N1—C2—C3	−0.8 (3)	N1—C6—C7—O4	−180.0 (2)
C6—N1—C2—C1	178.48 (19)	C5—C6—C7—O4	0.1 (4)
N1—C2—C3—C4	−0.5 (4)	N1—C6—C7—O3	−0.6 (3)
C1—C2—C3—C4	−179.8 (2)	C5—C6—C7—O3	179.4 (2)
C2—C3—C4—C5	1.1 (4)	C12—N2—C8—N3	−178.6 (3)
C3—C4—C5—C6	−0.4 (4)	C12—N2—C8—C9	0.4 (4)
C2—N1—C6—C5	1.5 (3)	N3—C8—C9—C10	178.9 (3)
C2—N1—C6—C7	−178.5 (2)	N2—C8—C9—C10	0.0 (4)
C4—C5—C6—N1	−0.9 (4)	C8—C9—C10—C11	−0.5 (4)
C4—C5—C6—C7	179.1 (2)	C8—C9—C10—C13	179.1 (2)
N1—C2—C1—O2	−172.4 (2)	C9—C10—C11—C12	0.5 (4)
C3—C2—C1—O2	6.9 (4)	C13—C10—C11—C12	−179.0 (3)
N1—C2—C1—O1	6.6 (3)	C10—C11—C12—N2	−0.1 (4)
C3—C2—C1—O1	−174.1 (2)	C8—N2—C12—C11	−0.3 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*C*···O2^{i}	0.96	2.59	3.488 (5)	157
O5—H5*A*···O3^{ii}	0.78 (5)	2.02 (5)	2.796 (3)	170 (4)
N3—H3*B*···O4^{iii}	0.83 (4)	1.95 (4)	2.764 (3)	166 (3)
N3—H3*A*···O2^{i}	0.87 (4)	2.30 (4)	3.122 (3)	158 (3)
N2—H2···O3^{iii}	0.85 (3)	1.87 (3)	2.723 (3)	173 (3)
O1—H1···O5^{iv}	0.87 (4)	1.87 (4)	2.689 (3)	156 (4)

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