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Pyridine-2,6-dicarbaldehyde bis(benzylidenehydrazone) monohydrate

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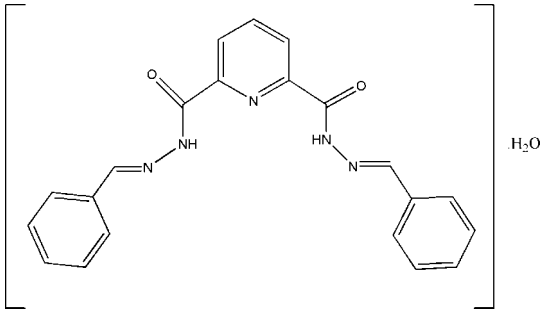
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.108; data-to-parameter ratio = 8.3.

The crystal structure of the title compound, $C_{21}H_{17}N_5O_2\cdot H_2O$, is stabilized by intermolecular hydrogen bonds.

Related literature

For related literature, see: Chen *et al.* (1997); Thompson (2002); Zhao *et al.* (2004).



Experimental

Crystal data

$C_{21}H_{17}N_5O_2\cdot H_2O$
M_r = 389.41
Orthorhombic, $Pna2_1$
a = 10.3490 (8) Å
b = 16.2487 (12) Å
c = 11.8556 (9) Å
V = 1993.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
11761 measured reflections
2278 independent reflections
1653 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.108
S = 0.98
2278 reflections
274 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O3	0.93	2.48	3.305 (5)	148
N2—H2A···O3	0.86 (4)	2.35 (4)	3.126 (4)	150 (4)
O3—H3B···O2^{i}	0.90 (4)	2.06 (4)	2.874 (3)	149 (4)
N4—H4A···O1^{ii}	0.87 (4)	2.32 (4)	3.044 (4)	142 (4)
O3—H3A···O1^{ii}	0.85 (5)	2.14 (5)	2.790 (4)	133 (5)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); 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: BT2363).

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supplementary materials

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Pyridine-2,6-dicarbaldehyde bis(benzylidenehydrazone) monohydrate

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Comment

Tridentate ligands containing the 2,6-dipicolinoyhydrazone moiety have been intensively studied due to their interesting coordination mode (Chen *et al.*, 1997; Thompson *et al.*,2002; Zhao *et al.*, 2004).

The molecules are linked into a three dimensional framework by a combination of N–H···O C—H···O and O—H···O hydrogen bonds.

Experimental

To a solution of benzaldehyde (1.166 g, 11 mmol) in absolute ethanol (40 ml) a suspension of 2,6-dipicolinoyhydrazine in the same solvent (50 ml) was added at 353 K. The mixture was left to react at reflux for 8 h. Then, the white product was filtered, washed with hot ethanol (20 ml) three times and dried in vacuo. Crystals suitable for X-ray diffraction were obtained from dimethylformamide–methanol (3:1 v/v) over a period of about three weeks.

Refinement

All H atoms were located in difference maps. H atoms bonded to C were then treated as riding with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The coordinates of the remaining H atoms were refined, but U_{iso}(H) was set to 1.2U_{eq}(N,*O*).

Figures

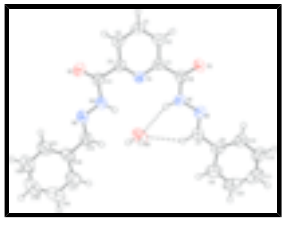


Fig. 1. The molecular structure of the title compound with atom labels and 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

Pyridine-2,6-dicarbaldehyde bis(benzylidenehydrazone) monohydrate

Crystal data

$C_{21}H_{17}N_5O_2·H_2O$	D_x = 1.297 Mg m^{-3}
M_r = 389.41	Mo *K*α radiation λ = 0.71073 Å
Orthorhombic, *Pna*2$_1$	Cell parameters from 1982 reflections
a = 10.3490 (8) Å	θ = 2.5–21.4°
b = 16.2487 (12) Å	μ = 0.09 mm^{-1}

c = 11.8556 (9) Å	T = 298 (2) K
V = 1993.6 (3) Å^3	Block, colourless
Z = 4	0.20 × 0.10 × 0.10 mm
F_{000} = 816	

Data collection

Bruker SMART CCD area-detector diffractometer	1653 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.077
Monochromator: graphite	θ_{max} = 27.0º
T = 298(2) K	θ_{min} = 2.1º
φ and ω scans	h = −9→13
Absorption correction: none	k = −20→20
11761 measured reflections	l = −12→15
2278 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.048	$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.108	$(\Delta/\sigma)_{max} < 0.001$
S = 0.98	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
2278 reflections	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
274 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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. 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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.1229 (3)	0.68779 (19)	0.7135 (3)	0.0417 (7)
C2	0.0307 (3)	0.6861 (2)	0.7975 (3)	0.0560 (9)
H2	−0.0476	0.7133	0.7885	0.067*
C3	0.0570 (4)	0.6437 (2)	0.8943 (3)	0.0674 (10)
H3	−0.0035	0.6416	0.9522	0.081*
C4	0.1735 (3)	0.6041 (2)	0.9053 (3)	0.0575 (9)
H4	0.1934	0.5748	0.9704	0.069*
C5	0.2607 (3)	0.60884 (18)	0.8169 (3)	0.0426 (7)
C6	0.0924 (3)	0.73123 (18)	0.6064 (3)	0.0456 (7)
C7	0.2320 (4)	0.7503 (2)	0.3423 (3)	0.0591 (10)
H7	0.3023	0.7157	0.3541	0.071*
C8	0.2124 (3)	0.7865 (2)	0.2314 (3)	0.0538 (8)
C9	0.2824 (4)	0.7571 (2)	0.1402 (3)	0.0693 (11)
H9	0.3439	0.7161	0.1512	0.083*
C10	0.2617 (4)	0.7881 (3)	0.0331 (3)	0.0757 (12)
H10	0.3082	0.7673	−0.0277	0.091*
C11	0.1732 (4)	0.8494 (3)	0.0162 (4)	0.0751 (12)
H11	0.1598	0.8704	−0.0558	0.090*
C12	0.1055 (4)	0.8790 (2)	0.1043 (3)	0.0689 (11)
H12	0.0455	0.9208	0.0923	0.083*
C13	0.1232 (3)	0.8489 (2)	0.2117 (3)	0.0597 (9)
H13	0.0753	0.8703	0.2712	0.072*
C14	0.3879 (3)	0.56512 (19)	0.8299 (3)	0.0463 (8)
C15	0.6689 (3)	0.5587 (2)	0.6706 (3)	0.0515 (8)
H15	0.6409	0.5909	0.6106	0.062*
C16	0.8009 (3)	0.5264 (2)	0.6703 (3)	0.0546 (9)
C17	0.8874 (3)	0.5537 (2)	0.5896 (3)	0.0621 (9)
H17	0.8617	0.5924	0.5365	0.075*
C18	1.0127 (4)	0.5230 (3)	0.5881 (4)	0.0775 (13)
H18	1.0708	0.5413	0.5337	0.093*
C19	1.0508 (4)	0.4663 (3)	0.6659 (5)	0.0834 (14)
H19	1.1349	0.4461	0.6649	0.100*
C20	0.9653 (4)	0.4391 (3)	0.7455 (5)	0.0863 (13)
H20	0.9915	0.4002	0.7982	0.104*
C21	0.8410 (3)	0.4687 (2)	0.7486 (4)	0.0705 (11)
H21	0.7837	0.4498	0.8032	0.085*
N1	0.2374 (2)	0.64974 (15)	0.7219 (2)	0.0418 (6)
N2	0.1809 (3)	0.72613 (19)	0.5239 (3)	0.0575 (8)
N3	0.1550 (2)	0.76508 (16)	0.4236 (2)	0.0553 (7)
N4	0.4704 (3)	0.57779 (18)	0.7442 (2)	0.0497 (7)
N5	0.5920 (2)	0.54337 (16)	0.7514 (2)	0.0514 (7)
O1	−0.0089 (2)	0.76993 (14)	0.5958 (2)	0.0605 (6)
O2	0.4109 (2)	0.52270 (15)	0.9118 (2)	0.0654 (7)
O3	0.3994 (3)	0.60103 (18)	0.4627 (3)	0.0724 (8)
H4A	0.454 (4)	0.605 (2)	0.683 (4)	0.087*

H2A	0.244 (4)	0.693 (3)	0.535 (4)	0.087*
H3B	0.431 (4)	0.551 (3)	0.445 (4)	0.109*
H3A	0.458 (4)	0.637 (3)	0.470 (5)	0.109*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0390 (16)	0.0443 (16)	0.0419 (18)	0.0012 (13)	0.0026 (13)	0.0021 (13)
C2	0.051 (2)	0.063 (2)	0.054 (2)	0.0104 (16)	0.0116 (17)	0.0085 (18)
C3	0.061 (2)	0.088 (3)	0.053 (2)	0.0149 (19)	0.0240 (18)	0.017 (2)
C4	0.064 (2)	0.064 (2)	0.044 (2)	0.0074 (17)	0.0107 (16)	0.0135 (17)
C5	0.0456 (18)	0.0419 (16)	0.0403 (18)	−0.0040 (13)	0.0027 (14)	0.0041 (14)
C6	0.0435 (17)	0.0439 (17)	0.0495 (19)	0.0039 (14)	0.0022 (15)	0.0026 (15)
C7	0.055 (2)	0.065 (2)	0.057 (2)	0.0169 (17)	0.0111 (17)	0.0182 (19)
C8	0.0539 (19)	0.057 (2)	0.051 (2)	0.0010 (16)	0.0118 (16)	0.0071 (17)
C9	0.075 (3)	0.066 (2)	0.067 (3)	−0.002 (2)	0.023 (2)	0.000 (2)
C10	0.092 (3)	0.085 (3)	0.049 (2)	−0.026 (3)	0.026 (2)	−0.014 (2)
C11	0.085 (3)	0.094 (3)	0.046 (2)	−0.025 (3)	−0.004 (2)	0.012 (2)
C12	0.067 (2)	0.085 (3)	0.055 (2)	0.0016 (19)	−0.004 (2)	0.018 (2)
C13	0.059 (2)	0.072 (2)	0.048 (2)	0.0052 (18)	0.0053 (17)	0.0113 (18)
C14	0.0490 (19)	0.0480 (18)	0.0418 (18)	−0.0008 (14)	−0.0016 (14)	0.0046 (15)
C15	0.0486 (19)	0.0551 (19)	0.051 (2)	0.0092 (15)	−0.0054 (16)	−0.0033 (17)
C16	0.0448 (18)	0.055 (2)	0.064 (2)	0.0056 (15)	−0.0024 (17)	−0.0187 (18)
C17	0.053 (2)	0.064 (2)	0.069 (3)	−0.0008 (17)	0.0032 (18)	−0.016 (2)
C18	0.056 (2)	0.084 (3)	0.093 (3)	−0.011 (2)	0.020 (2)	−0.034 (3)
C19	0.052 (2)	0.087 (3)	0.112 (4)	0.017 (2)	−0.012 (3)	−0.033 (3)
C20	0.062 (2)	0.094 (3)	0.102 (4)	0.024 (2)	−0.010 (3)	−0.003 (3)
C21	0.055 (2)	0.082 (3)	0.074 (3)	0.0168 (19)	0.003 (2)	−0.001 (2)
N1	0.0399 (13)	0.0447 (13)	0.0409 (15)	−0.0007 (11)	0.0034 (11)	0.0062 (12)
N2	0.0509 (17)	0.072 (2)	0.0499 (17)	0.0186 (14)	0.0112 (14)	0.0248 (15)
N3	0.0534 (16)	0.0626 (17)	0.0498 (18)	0.0096 (13)	0.0082 (14)	0.0172 (15)
N4	0.0430 (14)	0.0621 (17)	0.0441 (16)	0.0105 (12)	−0.0008 (12)	0.0084 (13)
N5	0.0440 (15)	0.0594 (16)	0.0508 (17)	0.0110 (12)	−0.0021 (13)	0.0029 (14)
O1	0.0526 (13)	0.0707 (15)	0.0581 (15)	0.0241 (11)	0.0065 (12)	0.0106 (13)
O2	0.0650 (15)	0.0791 (16)	0.0521 (15)	0.0141 (12)	0.0000 (11)	0.0228 (14)
O3	0.0643 (17)	0.0628 (16)	0.090 (2)	0.0070 (13)	0.0042 (14)	−0.0171 (15)

Geometric parameters (Å, °)

C1—N1	1.340 (4)	C12—H12	0.9300
C1—C2	1.380 (4)	C13—H13	0.9300
C1—C6	1.486 (4)	C14—O2	1.214 (4)
C2—C3	1.366 (5)	C14—N4	1.343 (4)
C2—H2	0.9300	C15—N5	1.271 (4)
C3—C4	1.373 (5)	C15—C16	1.463 (4)
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.385 (4)	C16—C17	1.383 (5)
C4—H4	0.9300	C16—C21	1.383 (5)
C5—N1	1.330 (4)	C17—C18	1.389 (5)

C5—C14	1.504 (4)	C17—H17	0.9300
C6—O1	1.229 (3)	C18—C19	1.363 (7)
C6—N2	1.343 (4)	C18—H18	0.9300
C7—N3	1.274 (4)	C19—C20	1.367 (7)
C7—C8	1.454 (5)	C19—H19	0.9300
C7—H7	0.9300	C20—C21	1.373 (5)
C8—C9	1.387 (5)	C20—H20	0.9300
C8—C13	1.391 (5)	C21—H21	0.9300
C9—C10	1.383 (6)	N2—N3	1.374 (4)
C9—H9	0.9300	N2—H2A	0.86 (4)
C10—C11	1.367 (6)	N4—N5	1.380 (3)
C10—H10	0.9300	N4—H4A	0.87 (4)
C11—C12	1.347 (6)	O3—H3B	0.90 (4)
C11—H11	0.9300	O3—H3A	0.85 (5)
C12—C13	1.376 (5)		
N1—C1—C2	123.2 (3)	C12—C13—H13	119.9
N1—C1—C6	118.1 (2)	C8—C13—H13	119.9
C2—C1—C6	118.6 (3)	O2—C14—N4	124.6 (3)
C3—C2—C1	118.6 (3)	O2—C14—C5	121.5 (3)
C3—C2—H2	120.7	N4—C14—C5	114.0 (3)
C1—C2—H2	120.7	N5—C15—C16	121.1 (3)
C2—C3—C4	119.4 (3)	N5—C15—H15	119.4
C2—C3—H3	120.3	C16—C15—H15	119.4
C4—C3—H3	120.3	C17—C16—C21	119.2 (3)
C3—C4—C5	118.3 (3)	C17—C16—C15	119.4 (3)
C3—C4—H4	120.9	C21—C16—C15	121.4 (3)
C5—C4—H4	120.9	C16—C17—C18	119.9 (4)
N1—C5—C4	123.4 (3)	C16—C17—H17	120.0
N1—C5—C14	118.8 (3)	C18—C17—H17	120.0
C4—C5—C14	117.8 (3)	C19—C18—C17	120.2 (4)
O1—C6—N2	122.6 (3)	C19—C18—H18	119.9
O1—C6—C1	120.8 (3)	C17—C18—H18	119.9
N2—C6—C1	116.6 (3)	C18—C19—C20	119.9 (4)
N3—C7—C8	121.4 (3)	C18—C19—H19	120.0
N3—C7—H7	119.3	C20—C19—H19	120.0
C8—C7—H7	119.3	C19—C20—C21	120.8 (5)
C9—C8—C13	117.8 (3)	C19—C20—H20	119.6
C9—C8—C7	119.5 (3)	C21—C20—H20	119.6
C13—C8—C7	122.6 (3)	C20—C21—C16	120.0 (4)
C10—C9—C8	120.6 (4)	C20—C21—H21	120.0
C10—C9—H9	119.7	C16—C21—H21	120.0
C8—C9—H9	119.7	C5—N1—C1	117.0 (2)
C11—C10—C9	120.3 (4)	C6—N2—N3	118.0 (3)
C11—C10—H10	119.9	C6—N2—H2A	117 (3)
C9—C10—H10	119.9	N3—N2—H2A	125 (3)
C12—C11—C10	119.7 (4)	C7—N3—N2	116.5 (3)
C12—C11—H11	120.2	C14—N4—N5	118.1 (3)
C10—C11—H11	120.2	C14—N4—H4A	126 (3)
C11—C12—C13	121.4 (4)	N5—N4—H4A	116 (3)

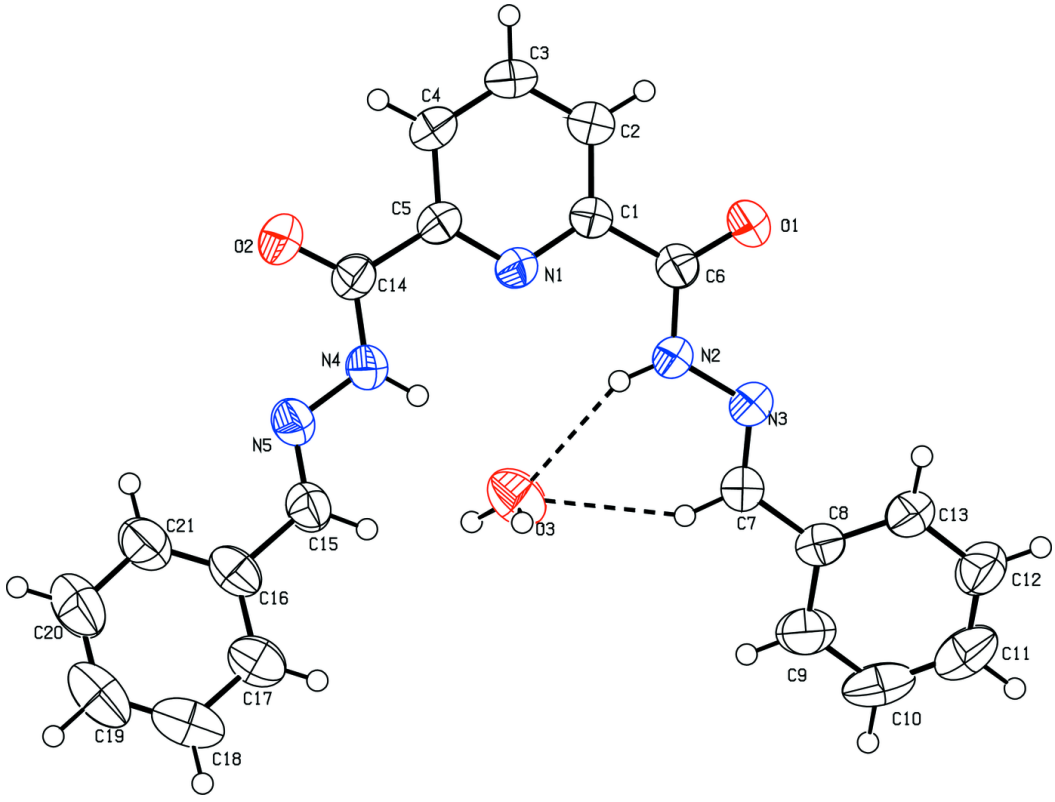
C11—C12—H12	119.3	C15—N5—N4	116.5 (3)
C13—C12—H12	119.3	H3B—O3—H3A	113 (4)
C12—C13—C8	120.2 (3)		
N1—C1—C2—C3	0.0 (5)	C4—C5—C14—N4	−175.4 (3)
C6—C1—C2—C3	177.6 (3)	N5—C15—C16—C17	−170.8 (3)
C1—C2—C3—C4	0.0 (6)	N5—C15—C16—C21	9.7 (5)
C2—C3—C4—C5	0.0 (6)	C21—C16—C17—C18	−0.1 (5)
C3—C4—C5—N1	0.0 (5)	C15—C16—C17—C18	−179.6 (3)
C3—C4—C5—C14	−179.9 (3)	C16—C17—C18—C19	−0.1 (6)
N1—C1—C6—O1	−177.0 (3)	C17—C18—C19—C20	0.3 (6)
C2—C1—C6—O1	5.2 (4)	C18—C19—C20—C21	−0.4 (7)
N1—C1—C6—N2	2.6 (4)	C19—C20—C21—C16	0.2 (7)
C2—C1—C6—N2	−175.2 (3)	C17—C16—C21—C20	0.0 (6)
N3—C7—C8—C9	−167.6 (4)	C15—C16—C21—C20	179.5 (4)
N3—C7—C8—C13	10.9 (6)	C4—C5—N1—C1	0.0 (4)
C13—C8—C9—C10	−1.2 (6)	C14—C5—N1—C1	179.9 (3)
C7—C8—C9—C10	177.3 (4)	C2—C1—N1—C5	0.0 (4)
C8—C9—C10—C11	1.1 (6)	C6—C1—N1—C5	−177.7 (3)
C9—C10—C11—C12	−0.4 (6)	O1—C6—N2—N3	−0.9 (5)
C10—C11—C12—C13	−0.1 (6)	C1—C6—N2—N3	179.5 (3)
C11—C12—C13—C8	0.0 (6)	C8—C7—N3—N2	178.5 (3)
C9—C8—C13—C12	0.7 (5)	C6—N2—N3—C7	−171.0 (3)
C7—C8—C13—C12	−177.8 (4)	O2—C14—N4—N5	−3.0 (5)
N1—C5—C14—O2	−175.3 (3)	C5—C14—N4—N5	176.9 (3)
C4—C5—C14—O2	4.6 (5)	C16—C15—N5—N4	179.0 (3)
N1—C5—C14—N4	4.7 (4)	C14—N4—N5—C15	−177.9 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O3	0.93	2.48	3.305 (5)	148
N2—H2A···O3	0.86 (4)	2.35 (4)	3.126 (4)	150 (4)
O3—H3B···O2^{i}	0.90 (4)	2.06 (4)	2.874 (3)	149 (4)
N4—H4A···O1^{ii}	0.87 (4)	2.32 (4)	3.044 (4)	142 (4)
O3—H3A···O1^{ii}	0.85 (5)	2.14 (5)	2.790 (4)	133 (5)

Symmetry codes: (i) −*x*+1, −*y*+1, *z*−1/2; (ii) *x*+1/2, −*y*+3/2, *z*.

Fig. 1


C3
C4
C2
C5
C1
O1
O2
C14
N1
C6
N4
N2
N5
N3
O3
C7
C8
C13
C21
C15
C16
C12
C20
C11
C9
C17
C10
C19
C18