organic compounds

Glutaric acid–2-(pyridin-4-yl)-1*H*-benzimidazole (1/1)

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The crystal structure of the title co-crystal, $C_{12}H_9N_3 \cdot C_5H_8O_4$, N—H···O and O—H···N hydrogen bonds link the components. There are also π–π stacking interactions between the imidazole rings, between the imidazole and pyridine rings and between the pyridine and benzene rings [centroid–centroid distances = 3.643 (2), 3.573 (2) and 3.740 (1)Å, respectively].

Related literature

For background to hydrogen bonds, see: Moorthy *et al.* (2002); Muthuraman *et al.* (2000); Nangia & Desiraju (1999); Bhattacharjya *et al.* (2004). For related structures, see: Bei *et al.* (2000); Ozbey *et al.* (1998).
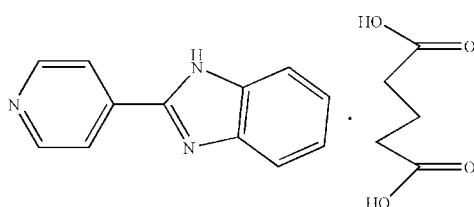
Experimental

Crystal data

$C_{12}H_9N_3 \cdot C_5H_8O_4$	$\gamma = 85.57\ (3)°$
$M_r = 327.34$	$V = 782.1\ (3)$ Å^3
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.4384\ (15)$ Å	Mo $K\alpha$ radiation
$b = 8.9911\ (18)$ Å	$\mu = 0.10$ mm^{-1}
$c = 11.868\ (2)$ Å	$T = 293$ K
$\alpha = 86.67\ (3)°$	$0.20 \times 0.17 \times 0.15$ mm
$\beta = 81.66\ (3)°$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.024$
6041 measured reflections	3 standard reflections every 100 reflections
2664 independent reflections	intensity decay: none
1657 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.149$	
$S = 1.11$	
2664 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
226 parameters	$\Delta\rho_{min} = -0.32$ e Å^{-3}
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^i	0.86	2.10	2.957 (3)	176
O1—H2···N3^{ii}	0.87 (1)	1.75 (1)	2.615 (3)	173 (4)
O4—H1···N2	1.02 (4)	1.71 (4)	2.686 (3)	158 (3)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); 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: *WinGX* (Farrugia, 1999).

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supporting information

Glutaric acid–2-(pyridin-4-yl)-1*H*-benzimidazole (1/1)

Songzhu Lin, Ruokun Jia, Aimin He and Xiaoli Gao

S1. Comment

The strong (O—H···O) and weak (C—H···O) hydrogen bonds, the halogen bond (C—*X*···O) and the weak C—H···π interaction, have been well characterized and exploited in the design of molecular assemblies (Moorthy *et al.*, 2002; Muthuraman *et al.*, 2000; Nangia and Desiraju, 1999; Bhattacharjya *et al.*, 2004). Our interest in benzimidazole stems from their biological activity (Bei *et al.*, 2000; Ozbey *et al.*, 1998). In this paper, we sysnthesized the title compound and report its structure.

Scheme I

The compound consists of 2-(pyridin-4-yl)-1*H*-benzimidazole and glutaric acid. In the title compound, the dihedral angle between the imidazole and the benzene was 1.40 (2)°, while the benzimidazole and the pyridine was 5.25 (1)°. It results that the all atoms in the 2-(pyridin-4-yl)-1*H*-benzimidazole are not coplanar strictly. In the part of glutaric acid, four atoms O1, O2, C13, C14 are lying in a same plane (p1) with the maximum diviation of 0.002° for C13, while other four atoms O3, O4, C16, C17 lying in another plane (p2) with the maximum diviation of 0.001% for O3. The dihedral angle between p1 and p2 is 10.50 (2)°.

In the lattice, there exist some kinds of hydrogen bonds. It forms one-dimension stairway structure between 2-(pyridin-4-yl)-1*H*-benzimidazole and glutaric acid *via* N—H···O, O—H···N hydrogen bonds (figure 2a and 2 b). Two adjacent strairway chains formed two dimension structure *via* the C—H···O intermolecular interaction.

In addition, there exists some π–π interactions between the rings [$Cg1···Cg1$=3.643 (2), $Cg1···Cg2$= 3.573 (2) and $Cg2···Cg3$=3.740 (1), respectively ($Cg1$, $Cg2$, $Cg3$ refer to the centroid of imidazole N1, C1, C6, N2, C7; the pyridine N3, C8, C9, C10, C11, C12 and the phenyl ring C1, C2, C3, C4, C5, C6, respectively)]. The π–π interaction, as well as the inter- and intra- hydrogen bond stabilized the crystal structure.

S2. Experimental

The title compound was obtained by 2-Pyridin-4-yl-1*H*-benzoimidazole (0.020 g, 0.1 mmol) and glutaric acid (0.013 g, 0.1 mmol) dissolved in 30 ml solution mixed with ethanol and water by 2:1(V/V) was heated to refluxed for 6 h and cooled to the room temperature. Single crystals suitable for *x*-ray measurements were obtained by recrystallization at room temperature.

S3. Refinement

The positions of H atoms, H1,H2, were found in a difference Fourier map. All the other H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances=0.93–0.97 Å, N—H distance=0.86Å and with U_{iso}=1.2–1.5U_{eq}.
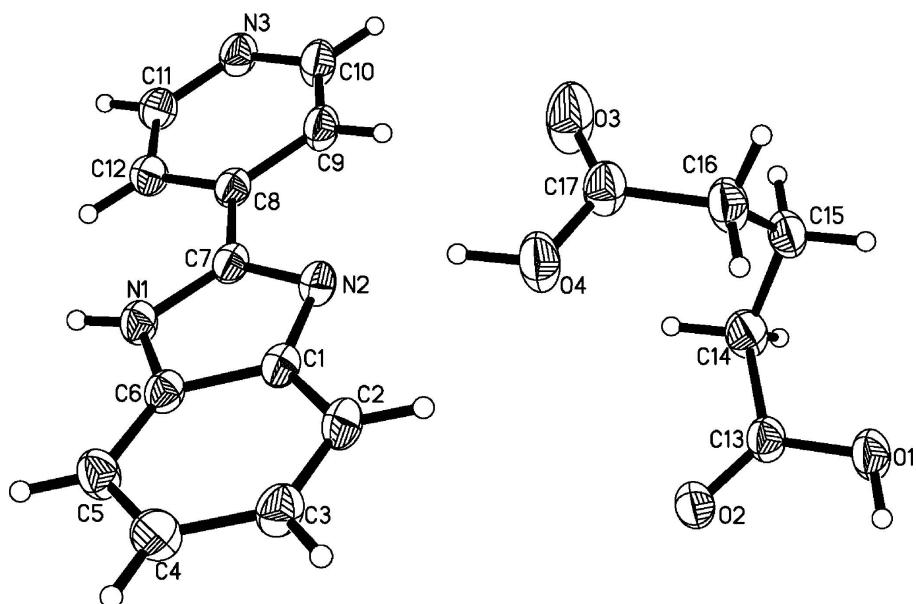
Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.
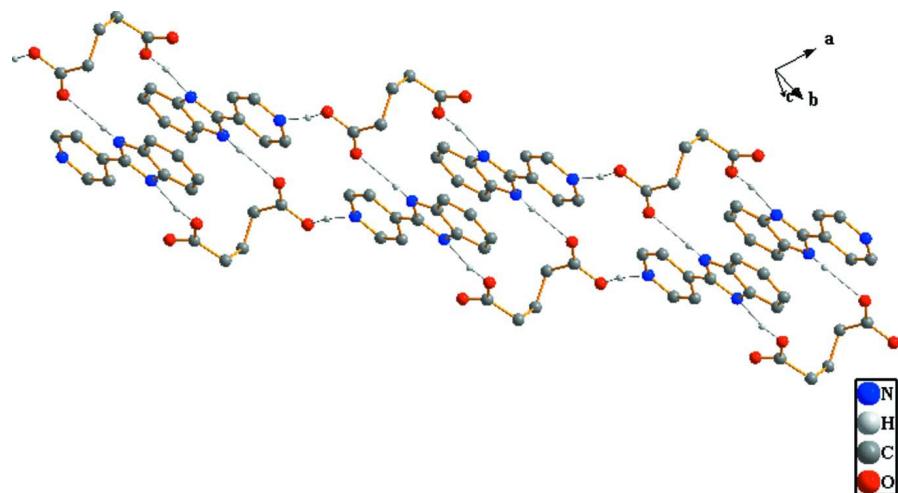
Figure 2

One-dimensional bend structure of the title compound.
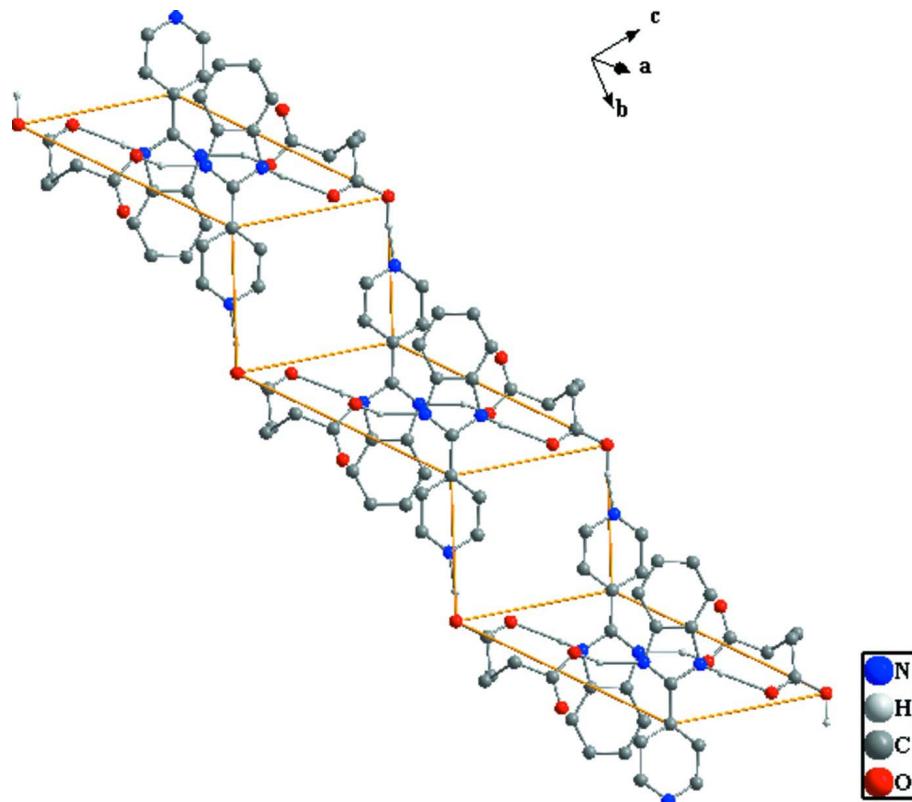
Figure 3

Two-dimensional structure of the title compound.

Pentane-1,5-dioic acid–2-(pyridin-4-yl)-1*H*-benzimidazole (1/1)

Crystal data

$C_{12}H_9N_3 \cdot C_5H_8O_4$	$Z = 2$
$M_r = 327.34$	$F(000) = 344$
Triclinic, $P\bar{1}$	$D_x = 1.390$ Mg m^{-3}
Hall symbol: -p 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.4384$ (15) Å	Cell parameters from 25 reflections
$b = 8.9911$ (18) Å	$\theta = 4$–14°
$c = 11.868$ (2) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 86.67$ (3)°	$T = 293$ K
$\beta = 81.66$ (3)°	Block, colorless
$\gamma = 85.57$ (3)°	$0.20 \times 0.17 \times 0.15$ mm
$V = 782.1$ (3) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.024$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.0°$, $\theta_{min} = 3.1°$
Graphite monochromator	$h = -7 \rightarrow 7$
ω scans	$k = -10 \rightarrow 10$
6041 measured reflections	$l = -14 \rightarrow 13$
2664 independent reflections	3 standard reflections every 100 reflections
1657 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.149$	
$S = 1.11$	$w = 1/[\sigma^2(F_o^2) + (0.0825P)^2 + 0.020P]$ where $P = (F_o^2 + 2F_c^2)/3$
2664 reflections	
226 parameters	$(\Delta/\sigma)_{max} < 0.001$
1 restraint	$\Delta\rho_{max} = 0.35$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.32$ e Å^{-3}
	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Secondary atom site location: difference Fourier map	
	Extinction coefficient: 0.018 (5)

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}
N1	0.2659 (2)	0.5349 (2)	0.58023 (17)	0.0435 (5)
H1A	0.2940	0.6038	0.6206	0.052*
N2	0.2164 (2)	0.4197 (2)	0.42719 (17)	0.0432 (5)
N3	0.4250 (3)	0.9193 (2)	0.24990 (19)	0.0526 (6)
C1	0.1814 (3)	0.3243 (2)	0.5230 (2)	0.0403 (6)
C2	0.1261 (3)	0.1787 (3)	0.5328 (2)	0.0478 (6)
H2B	0.1037	0.1309	0.4693	0.057*
C3	0.1057 (3)	0.1083 (3)	0.6392 (2)	0.0515 (7)
H3B	0.0690	0.0112	0.6477	0.062*
C4	0.1387 (3)	0.1791 (3)	0.7345 (2)	0.0540 (7)
H4A	0.1251	0.1275	0.8051	0.065*
C5	0.1912 (3)	0.3235 (3)	0.7273 (2)	0.0508 (7)
H5A	0.2117	0.3707	0.7915	0.061*
C6	0.2117 (3)	0.3952 (3)	0.6200 (2)	0.0408 (6)
C7	0.2664 (3)	0.5429 (2)	0.4655 (2)	0.0405 (6)
C8	0.3189 (3)	0.6750 (3)	0.3922 (2)	0.0416 (6)
C9	0.3303 (3)	0.6703 (3)	0.2746 (2)	0.0502 (7)
H9A	0.3025	0.5851	0.2416	0.060*
C10	0.3835 (3)	0.7942 (3)	0.2073 (2)	0.0563 (7)
H10A	0.3908	0.7903	0.1286	0.068*
C11	0.4129 (3)	0.9229 (3)	0.3626 (2)	0.0520 (7)
H11A	0.4408	1.0097	0.3934	0.062*
C12	0.3614 (3)	0.8050 (3)	0.4361 (2)	0.0488 (6)

H12A	0.3553	0.8125	0.5144	0.059*
O1	−0.3978 (3)	0.1206 (2)	0.12019 (17)	0.0654 (6)
O2	−0.3751 (2)	0.2383 (2)	0.27704 (16)	0.0599 (5)
O3	0.2390 (4)	0.3965 (3)	0.1101 (2)	0.1041 (9)
O4	0.1264 (3)	0.2629 (2)	0.26029 (18)	0.0776 (7)
C13	−0.3421 (3)	0.2273 (3)	0.1748 (2)	0.0490 (6)
C14	−0.2312 (4)	0.3340 (3)	0.0967 (2)	0.0618 (8)
H14A	−0.1852	0.4040	0.1429	0.074*
H14B	−0.3110	0.3908	0.0497	0.074*
C15	−0.0728 (4)	0.2637 (3)	0.0197 (2)	0.0597 (7)
H15A	−0.0195	0.3404	−0.0327	0.072*
H15B	−0.1176	0.1914	−0.0252	0.072*
C16	0.0758 (4)	0.1854 (3)	0.0833 (2)	0.0619 (8)
H16A	0.0244	0.1063	0.1340	0.074*
H16B	0.1714	0.1405	0.0288	0.074*
C17	0.1557 (4)	0.2930 (3)	0.1513 (3)	0.0601 (8)
H1	0.173 (5)	0.338 (4)	0.308 (3)	0.123 (13)*
H2	−0.456 (4)	0.058 (4)	0.167 (3)	0.133 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0502 (11)	0.0392 (11)	0.0437 (13)	−0.0103 (8)	−0.0095 (9)	−0.0083 (9)
N2	0.0464 (11)	0.0400 (12)	0.0465 (13)	−0.0084 (9)	−0.0127 (9)	−0.0063 (10)
N3	0.0573 (12)	0.0472 (13)	0.0568 (15)	−0.0157 (10)	−0.0133 (10)	−0.0024 (11)
C1	0.0380 (12)	0.0384 (13)	0.0466 (15)	−0.0042 (9)	−0.0102 (10)	−0.0064 (11)
C2	0.0524 (14)	0.0402 (14)	0.0547 (17)	−0.0087 (11)	−0.0144 (11)	−0.0120 (12)
C3	0.0589 (15)	0.0391 (14)	0.0592 (18)	−0.0121 (11)	−0.0122 (12)	−0.0039 (13)
C4	0.0646 (16)	0.0458 (15)	0.0515 (17)	−0.0079 (12)	−0.0054 (13)	−0.0029 (13)
C5	0.0653 (16)	0.0461 (15)	0.0427 (16)	−0.0088 (12)	−0.0079 (12)	−0.0089 (12)
C6	0.0403 (12)	0.0364 (13)	0.0472 (15)	−0.0047 (9)	−0.0075 (10)	−0.0084 (11)
C7	0.0375 (12)	0.0408 (13)	0.0454 (15)	−0.0066 (10)	−0.0098 (10)	−0.0061 (11)
C8	0.0387 (12)	0.0406 (13)	0.0475 (15)	−0.0064 (9)	−0.0089 (10)	−0.0071 (11)
C9	0.0582 (15)	0.0474 (15)	0.0499 (16)	−0.0168 (11)	−0.0160 (12)	−0.0054 (12)
C10	0.0678 (17)	0.0572 (17)	0.0500 (17)	−0.0221 (13)	−0.0207 (13)	0.0012 (13)
C11	0.0566 (15)	0.0448 (15)	0.0569 (18)	−0.0151 (11)	−0.0064 (12)	−0.0102 (13)
C12	0.0523 (14)	0.0485 (15)	0.0470 (16)	−0.0128 (11)	−0.0049 (11)	−0.0082 (12)
O1	0.0996 (15)	0.0540 (12)	0.0469 (12)	−0.0322 (11)	−0.0076 (10)	−0.0088 (10)
O2	0.0784 (12)	0.0601 (12)	0.0441 (12)	−0.0230 (9)	−0.0063 (9)	−0.0081 (9)
O3	0.170 (2)	0.0867 (17)	0.0689 (16)	−0.0736 (17)	−0.0346 (15)	0.0149 (13)
O4	0.1193 (18)	0.0732 (15)	0.0487 (14)	−0.0464 (13)	−0.0185 (12)	−0.0038 (11)
C13	0.0623 (15)	0.0415 (14)	0.0453 (17)	−0.0102 (11)	−0.0097 (12)	−0.0044 (12)
C14	0.090 (2)	0.0469 (16)	0.0497 (17)	−0.0231 (14)	−0.0054 (15)	−0.0005 (13)
C15	0.0806 (18)	0.0623 (18)	0.0390 (16)	−0.0253 (14)	−0.0061 (13)	−0.0068 (13)
C16	0.088 (2)	0.0522 (17)	0.0492 (18)	−0.0181 (14)	−0.0124 (14)	−0.0107 (14)
C17	0.0833 (19)	0.0498 (17)	0.0520 (19)	−0.0205 (14)	−0.0195 (14)	0.0043 (14)

Geometric parameters (Å, º)

N1—C7	1.359 (3)	C9—H9A	0.9300
N1—C6	1.385 (3)	C10—H10A	0.9300
N1—H1A	0.8600	C11—C12	1.375 (3)
N2—C7	1.320 (3)	C11—H11A	0.9300
N2—C1	1.390 (3)	C12—H12A	0.9300
N3—C11	1.330 (3)	O1—C13	1.313 (3)
N3—C10	1.334 (3)	O1—H2	0.866 (10)
C1—C2	1.396 (3)	O2—C13	1.211 (3)
C1—C6	1.402 (3)	O3—C17	1.199 (3)
C2—C3	1.373 (3)	O4—C17	1.296 (3)
C2—H2B	0.9300	O4—H1	1.02 (4)
C3—C4	1.390 (4)	C13—C14	1.503 (3)
C3—H3B	0.9300	C14—C15	1.504 (4)
C4—C5	1.379 (4)	C14—H14A	0.9700
C4—H4A	0.9300	C14—H14B	0.9700
C5—C6	1.387 (4)	C15—C16	1.532 (4)
C5—H5A	0.9300	C15—H15A	0.9700
C7—C8	1.475 (3)	C15—H15B	0.9700
C8—C12	1.383 (3)	C16—C17	1.503 (4)
C8—C9	1.389 (4)	C16—H16A	0.9700
C9—C10	1.381 (3)	C16—H16B	0.9700
C7—N1—C6	107.18 (19)	C9—C10—H10A	118.5
C7—N1—H1A	126.4	N3—C11—C12	123.4 (2)
C6—N1—H1A	126.4	N3—C11—H11A	118.3
C7—N2—C1	105.23 (19)	C12—C11—H11A	118.3
C11—N3—C10	117.5 (2)	C11—C12—C8	119.2 (2)
N2—C1—C2	130.2 (2)	C11—C12—H12A	120.4
N2—C1—C6	109.62 (19)	C8—C12—H12A	120.4
C2—C1—C6	120.2 (2)	C13—O1—H2	111 (3)
C3—C2—C1	117.8 (2)	C17—O4—H1	114 (2)
C3—C2—H2B	121.1	O2—C13—O1	123.9 (2)
C1—C2—H2B	121.1	O2—C13—C14	123.5 (2)
C2—C3—C4	121.4 (2)	O1—C13—C14	112.6 (2)
C2—C3—H3B	119.3	C13—C14—C15	115.5 (2)
C4—C3—H3B	119.3	C13—C14—H14A	108.4
C5—C4—C3	121.9 (2)	C15—C14—H14A	108.4
C5—C4—H4A	119.0	C13—C14—H14B	108.4
C3—C4—H4A	119.0	C15—C14—H14B	108.4
C4—C5—C6	116.9 (2)	H14A—C14—H14B	107.5
C4—C5—H5A	121.6	C14—C15—C16	113.8 (2)
C6—C5—H5A	121.6	C14—C15—H15A	108.8
N1—C6—C5	133.1 (2)	C16—C15—H15A	108.8
N1—C6—C1	105.2 (2)	C14—C15—H15B	108.8
C5—C6—C1	121.7 (2)	C16—C15—H15B	108.8
N2—C7—N1	112.81 (19)	H15A—C15—H15B	107.7

N2—C7—C8	124.0 (2)	C17—C16—C15	111.4 (2)
N1—C7—C8	123.2 (2)	C17—C16—H16A	109.3
C12—C8—C9	117.8 (2)	C15—C16—H16A	109.3
C12—C8—C7	122.3 (2)	C17—C16—H16B	109.3
C9—C8—C7	119.9 (2)	C15—C16—H16B	109.3
C10—C9—C8	118.9 (2)	H16A—C16—H16B	108.0
C10—C9—H9A	120.5	O3—C17—O4	122.6 (3)
C8—C9—H9A	120.5	O3—C17—C16	124.1 (3)
N3—C10—C9	123.1 (3)	O4—C17—C16	113.3 (2)
N3—C10—H10A	118.5		
C7—N2—C1—C2	−179.2 (2)	N2—C7—C8—C12	176.6 (2)
C7—N2—C1—C6	0.2 (2)	N1—C7—C8—C12	−4.0 (3)
N2—C1—C2—C3	178.4 (2)	N2—C7—C8—C9	−4.7 (3)
C6—C1—C2—C3	−1.0 (3)	N1—C7—C8—C9	174.8 (2)
C1—C2—C3—C4	0.0 (4)	C12—C8—C9—C10	0.2 (4)
C2—C3—C4—C5	0.8 (4)	C7—C8—C9—C10	−178.6 (2)
C3—C4—C5—C6	−0.7 (4)	C11—N3—C10—C9	−0.3 (4)
C7—N1—C6—C5	178.0 (2)	C8—C9—C10—N3	0.0 (4)
C7—N1—C6—C1	0.0 (2)	C10—N3—C11—C12	0.4 (4)
C4—C5—C6—N1	−178.0 (2)	N3—C11—C12—C8	−0.2 (4)
C4—C5—C6—C1	−0.2 (3)	C9—C8—C12—C11	−0.1 (3)
N2—C1—C6—N1	−0.1 (2)	C7—C8—C12—C11	178.7 (2)
C2—C1—C6—N1	179.4 (2)	O2—C13—C14—C15	126.6 (3)
N2—C1—C6—C5	−178.4 (2)	O1—C13—C14—C15	−52.9 (3)
C2—C1—C6—C5	1.1 (3)	C13—C14—C15—C16	−64.9 (3)
C1—N2—C7—N1	−0.2 (2)	C14—C15—C16—C17	−60.6 (3)
C1—N2—C7—C8	179.28 (19)	C15—C16—C17—O3	−64.2 (4)
C6—N1—C7—N2	0.2 (2)	C15—C16—C17—O4	116.1 (3)
C6—N1—C7—C8	−179.33 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2[i]	0.86	2.10	2.957 (3)	176
O1—H2···N3[ii]	0.87 (1)	1.75 (1)	2.615 (3)	173 (4)
O4—H1···N2	1.02 (4)	1.71 (4)	2.686 (3)	158 (3)

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