Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 3-[(1*H*-benzimidazol-1-yl)-methyl]-1-methyl-4-(4-methylphenyl)-2′-oxopyrrolidine-2-spiro-3′-1-benzimidazole-3-carboxylate

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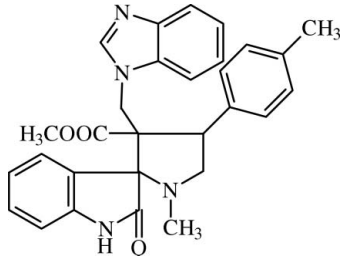
Received 27 August 2010; accepted 1 September 2010

Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.131; data-to-parameter ratio = 17.4.

In the title compound, $C_{29}H_{28}N_4O_3$, the pyrrolidine ring adopts a twist conformation whereas the oxindole and benzimidazole residues are approximately planar with maximum deviations of 0.159 (1) and 0.011 (1) Å, respectively. The oxindole residue is almost perpendicular to the benzimidazole residue, making a dihedral angle of 89.2 (1)°. The methyl-substituted benzene ring is oriented at angles of 47.7 (1) and 71.0 (1)°, respectively, with respect to the oxindole and benzimidazole residues. An intramolecular C—H···O hydrogen bond is observed. In the crystal, molecules associate *via* N—H···N hydrogen bonds, forming $R_2^2(9)$ dimers.

Related literature

For general background to pyrrolidine derivatives, see: Obniska *et al.* (2010); Morais *et al.* (2009); Bello *et al.* (2010); Moreno-Clavijo *et al.* (2009); Cheng *et al.* (2008). For related structures, see: Aravindan *et al.* (2004); Selvanayagam *et al.* (2005); Seshadri *et al.* (2003). For ring-puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{29}H_{28}N_4O_3$
M_r = 480.55
Triclinic, $P\overline{1}$
a = 9.7605 (5) Å
b = 11.2823 (6) Å
c = 12.2333 (7) Å
α = 79.960 (1)°
β = 69.539 (1)°
γ = 85.358 (1)°
V = 1242.53 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 292 K
0.26 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
14287 measured reflections
5705 independent reflections
5041 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.131
S = 1.04
5705 reflections
328 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 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
N2—H2···N4^{i}	0.86	2.05	2.888 (2)	165
C16—H16···O1	0.93	2.33	3.050 (2)	134

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

S*S* acknowledges the Department of Science and Technology (DST), India, for providing computing facilities under DST-Fast Track Scheme. S*S* also thanks the Vice Chancellor and management of Kalasalingam University, Krishnankoil, for their support and encouragement.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5341).

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supporting information

Acta Cryst. (2010). E**66**, o2508–o2509 [doi:10.1107/S1600536810035312]

Methyl 3-[(1*H*-benzimidazol-1-yl)methyl]-1-methyl-4-(4-methylphenyl)-2′-oxopyrrolidine-2-spiro-3′-1-benzimidazole-3-carboxylate

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S1. Comment

Pyrrolidine derivatives possess anticonvulsant (Obniska *et al.*, 2010), anti-angiogenic (Morais *et al.*, 2009) and antitumor (Bello *et al.*, 2010) activities. These derivatives are used as inhibitors of alpha-L-fucosidases (Moreno-Clavijo *et al.*, 2009) and matrix metalloproteinase (Cheng *et al.*, 2008). In view of these importance, we have undertaken the crystal structure determination of the title compound, a pyrrolidine derivative, and the results are presented here.

The molecular structure of (I) is illustrated in Fig. 1. The geometry of the pyrrolidine and oxindole residues of (I) compares well with that reported in other related structures (see, for example, Aravindan *et al.*, 2004; Selvanayagam *et al.*, 2005; Seshadri *et al.*, 2003).

The sum of the angles at N1 of the pyrrolidine ring [334.8°] and N3 of the imidazole ring [359.9°] are in accordance with sp^3 and sp^2 hybridizations. Atom O1 is essentially coplanar with the heterocyclic ring to which it is attached, with a deviation of -0.159 (1) Å. Benzimidazole residue is planar with a maximum deviation of -0.011 (1) Å for atom C22. Atom C29 deviates 0.052 (2) Å from the best plane of the methylphenyl ring.

The dihedral angle between the oxindole and benzimidazole residues is 89.2 (1)°. This indicates that the oxindole residue is almost perpendicular to the benzimidazole residue. The methyl phenyl ring is oriented at an angles of 47.7 (1) and 71.0 (1) ° with respect to the oxindole and benzimidazole residues.

The pyrrolidine ring adopts a twist conformation, with puckering parameters (Cremer & Pople, 1975) q_2 = 0.454 (1) Å and φ = 149.9 (2)°.

The molecular structure is influenced by an intramolecular C—H···O hydrogen bonds. Atom O1 acts as a bifurcated acceptor for two intramolecular C—H···O hydrogen bonds. In the molecular packing, N—H···N hydrogen bonds link inversion-related molecules to form $R_2^2(9)$ graph-set dimer (Fig.2 and Table 1).

S2. Experimental

To a mixture of isatin (1mmol), sarcosine (1mmol) and Baylis-Hillman adduct (1mmol) was added and heated under reflux in methanol (20ml) until the disappearance of the starting materials as evidenced by TLC. The solvent was removed under vacuo. The crude product was subjected to column chromatography using petroleum ether-ethyl acetate as eluent. Single crystals were grown by slow evaporation from methanol.

S3. Refinement

H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H distances of 0.93-0.97 Å and an N—H distance of 0.86 Å, and Uiso(H) = $1.5U_{eq}$(C) for methyl H and Uiso(H) = $1.2U_{eq}$(C,N) for all other H atoms.

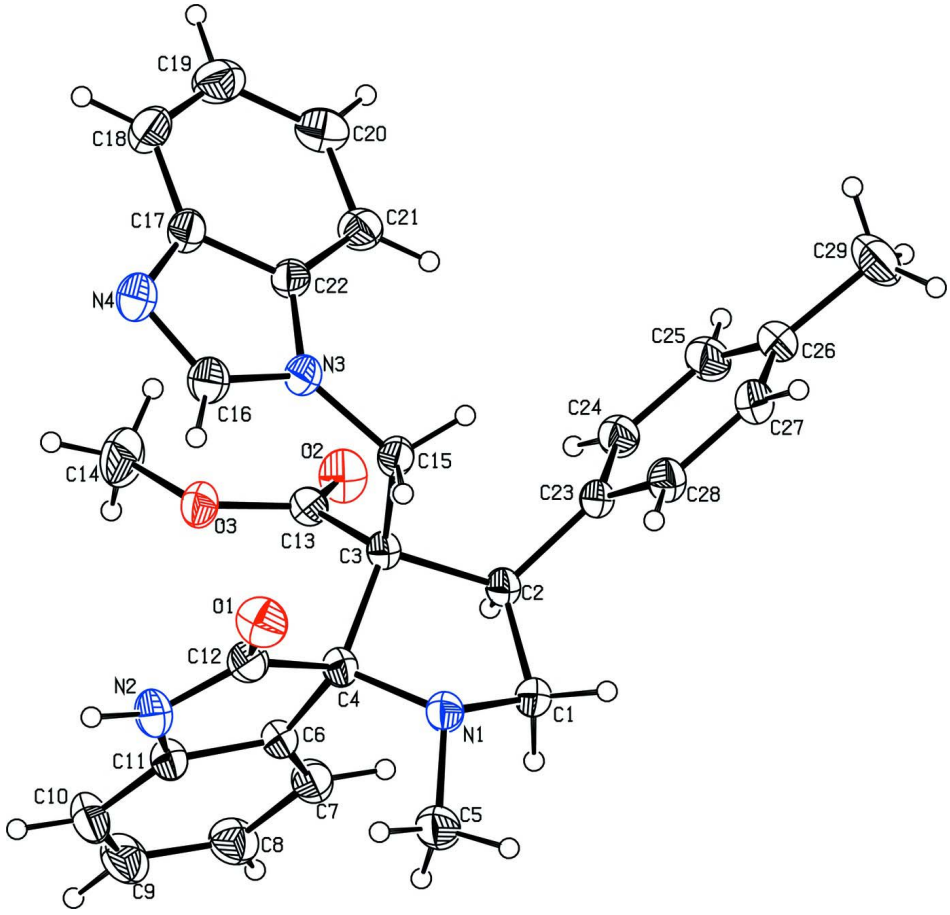

Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level

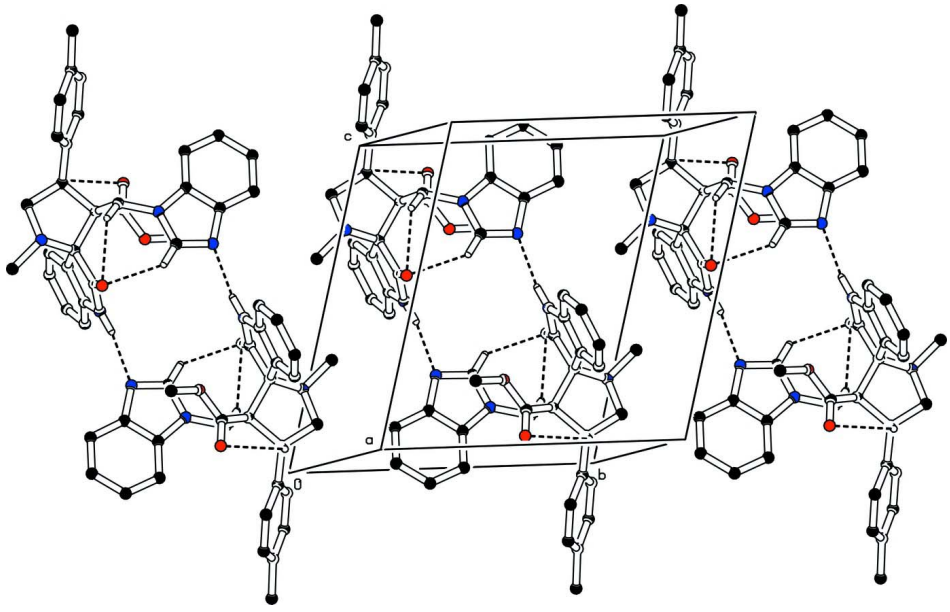

Figure 2

Molecular packing of the title compound, viewed down the *a* axis; H-bonds are shown as dashed lines. For the sake of clarity, H atoms, not involved in hydrogen bonds, have been omitted

Methyl 3-[(1*H*-benzimidazol-1-yl)methyl]-1-methyl-4-(4-methylphenyl)-2′- oxopyrrolidine-2-spiro-3′-1-benzimidazole-3-carboxylate

Crystal data

$C_{29}H_{28}N_4O_3$
$M_r = 480.55$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.7605$ (5) Å
$b = 11.2823$ (6) Å
$c = 12.2333$ (7) Å
$\alpha = 79.960$ (1)°
$\beta = 69.539$ (1)°
$\gamma = 85.358$ (1)°
$V = 1242.53$ (12) Å^3
$Z = 2$
$F(000) = 508$
$D_x = 1.284$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8628 reflections
$\theta = 2.0$–27.4°
$\mu = 0.09$ mm^{-1}
$T = 292$ K
Block, colourless
0.26 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
14287 measured reflections
5705 independent reflections
5041 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 28.0°$, $\theta_{min} = 1.8°$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.131$
$S = 1.04$
5705 reflections
328 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
$w = 1/[\sigma^2(F_o^2) + (0.0777P)^2 + 0.1893P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

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 $F^2 > 2$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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.02562 (10)	0.75938 (10)	0.40536 (9)	0.0564 (2)
O2	0.45463 (10)	0.62452 (9)	0.06224 (9)	0.0556 (2)
O3	0.30370 (10)	0.55640 (7)	0.24567 (8)	0.0475 (2)
N1	0.19339 (11)	0.93029 (8)	0.24498 (9)	0.0415 (2)
N2	0.16540 (13)	0.69375 (10)	0.47180 (9)	0.0498 (3)
H2	0.1161	0.6547	0.5395	0.060*
N3	0.05799 (10)	0.60212 (9)	0.17842 (8)	0.0392 (2)
N4	−0.04307 (15)	0.42908 (11)	0.28856 (10)	0.0592 (3)
C1	0.30089 (16)	0.97153 (11)	0.12846 (10)	0.0467 (3)
H1A	0.3882	0.9989	0.1363	0.056*
H1B	0.2603	1.0371	0.0857	0.056*
C2	0.33690 (12)	0.86094 (10)	0.06345 (9)	0.0371 (2)
H2A	0.4416	0.8431	0.0465	0.045*
C3	0.25260 (11)	0.75294 (9)	0.15982 (9)	0.0337 (2)
C4	0.23182 (12)	0.80336 (10)	0.27776 (9)	0.0363 (2)
C5	0.18078 (18)	1.00891 (12)	0.33182 (12)	0.0546 (3)
H5A	0.2735	1.0115	0.3421	0.082*
H5B	0.1087	0.9780	0.4059	0.082*
H5C	0.1518	1.0886	0.3043	0.082*
C6	0.36486 (13)	0.77598 (10)	0.31879 (10)	0.0383 (2)
C7	0.51257 (14)	0.80220 (12)	0.26535 (11)	0.0468 (3)
H7	0.5466	0.8478	0.1905	0.056*
C8	0.60970 (16)	0.75910 (14)	0.32568 (14)	0.0560 (3)
H8	0.7085	0.7767	0.2906	0.067*
C9	0.55981 (18)	0.69048 (15)	0.43698 (14)	0.0625 (4)

H9	0.6259	0.6620	0.4753	0.075*
C10	0.41285 (18)	0.66350 (14)	0.49225 (13)	0.0592 (4)
H10	0.3794	0.6173	0.5669	0.071*
C11	0.31760 (14)	0.70791 (11)	0.43221 (10)	0.0438 (3)
C12	0.10539 (13)	0.74875 (11)	0.39100 (10)	0.0427 (3)
C13	0.35074 (12)	0.63904 (10)	0.14798 (10)	0.0380 (2)
C14	0.3828 (2)	0.44195 (14)	0.24091 (17)	0.0713 (5)
H14A	0.3969	0.4160	0.1670	0.107*
H14B	0.3277	0.3831	0.3046	0.107*
H14C	0.4762	0.4509	0.2480	0.107*
C15	0.10345 (12)	0.72765 (10)	0.14655 (10)	0.0384 (2)
H15A	0.0272	0.7758	0.1952	0.046*
H15B	0.1105	0.7551	0.0651	0.046*
C16	−0.02908 (15)	0.54492 (13)	0.28527 (11)	0.0517 (3)
H16	−0.0745	0.5843	0.3499	0.062*
C17	0.04075 (15)	0.40654 (11)	0.17515 (12)	0.0481 (3)
C18	0.06481 (18)	0.29959 (12)	0.12571 (15)	0.0613 (4)
H18	0.0230	0.2278	0.1704	0.074*
C19	0.15226 (18)	0.30483 (13)	0.00910 (16)	0.0616 (4)
H19	0.1696	0.2351	−0.0251	0.074*
C20	0.21573 (15)	0.41222 (14)	−0.05946 (14)	0.0554 (3)
H20	0.2742	0.4120	−0.1379	0.066*
C21	0.19363 (13)	0.51890 (12)	−0.01339 (11)	0.0461 (3)
H21	0.2356	0.5903	−0.0590	0.055*
C22	0.10513 (12)	0.51376 (10)	0.10505 (10)	0.0390 (2)
C23	0.31253 (12)	0.88215 (10)	−0.05502 (9)	0.0361 (2)
C24	0.41258 (14)	0.83543 (11)	−0.15145 (11)	0.0440 (3)
H24	0.4914	0.7881	−0.1418	0.053*
C25	0.39601 (17)	0.85871 (13)	−0.26221 (11)	0.0536 (3)
H25	0.4643	0.8269	−0.3252	0.064*
C26	0.27911 (17)	0.92865 (12)	−0.27996 (11)	0.0505 (3)
C27	0.17746 (15)	0.97312 (11)	−0.18349 (11)	0.0475 (3)
H27	0.0969	1.0180	−0.1927	0.057*
C28	0.19461 (13)	0.95146 (11)	−0.07328 (10)	0.0428 (3)
H28	0.1263	0.9837	−0.0106	0.051*
C29	0.2617 (3)	0.95730 (18)	−0.40035 (15)	0.0789 (5)
H29A	0.3561	0.9562	−0.4609	0.118*
H29B	0.2167	1.0357	−0.4086	0.118*
H29C	0.2013	0.8982	−0.4077	0.118*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0441 (5)	0.0703 (6)	0.0480 (5)	−0.0038 (4)	−0.0057 (4)	−0.0113 (4)
O2	0.0484 (5)	0.0570 (5)	0.0528 (5)	0.0094 (4)	−0.0084 (4)	−0.0094 (4)
O3	0.0602 (5)	0.0349 (4)	0.0466 (5)	0.0031 (4)	−0.0212 (4)	0.0000 (3)
N1	0.0536 (6)	0.0351 (5)	0.0368 (5)	0.0028 (4)	−0.0177 (4)	−0.0051 (4)
N2	0.0576 (6)	0.0524 (6)	0.0318 (5)	−0.0095 (5)	−0.0096 (4)	0.0048 (4)

N3	0.0406 (5)	0.0383 (5)	0.0365 (5)	−0.0090 (4)	−0.0115 (4)	0.0004 (4)
N4	0.0737 (8)	0.0534 (6)	0.0456 (6)	−0.0246 (6)	−0.0172 (6)	0.0090 (5)
C1	0.0694 (8)	0.0361 (6)	0.0368 (6)	−0.0109 (5)	−0.0216 (5)	0.0000 (5)
C2	0.0427 (5)	0.0368 (5)	0.0314 (5)	−0.0079 (4)	−0.0135 (4)	0.0008 (4)
C3	0.0368 (5)	0.0332 (5)	0.0298 (5)	−0.0030 (4)	−0.0112 (4)	−0.0009 (4)
C4	0.0406 (5)	0.0359 (5)	0.0307 (5)	−0.0023 (4)	−0.0114 (4)	−0.0018 (4)
C5	0.0766 (9)	0.0460 (7)	0.0448 (7)	0.0063 (6)	−0.0237 (6)	−0.0134 (5)
C6	0.0470 (6)	0.0363 (5)	0.0331 (5)	−0.0020 (4)	−0.0163 (4)	−0.0036 (4)
C7	0.0483 (6)	0.0503 (7)	0.0425 (6)	−0.0057 (5)	−0.0179 (5)	−0.0016 (5)
C8	0.0509 (7)	0.0625 (8)	0.0613 (8)	0.0006 (6)	−0.0275 (6)	−0.0106 (7)
C9	0.0697 (9)	0.0665 (9)	0.0634 (9)	0.0109 (7)	−0.0415 (8)	−0.0080 (7)
C10	0.0788 (10)	0.0575 (8)	0.0446 (7)	0.0028 (7)	−0.0319 (7)	0.0039 (6)
C11	0.0566 (7)	0.0398 (6)	0.0351 (6)	−0.0025 (5)	−0.0175 (5)	−0.0017 (4)
C12	0.0463 (6)	0.0436 (6)	0.0343 (5)	−0.0047 (5)	−0.0075 (5)	−0.0070 (4)
C13	0.0406 (5)	0.0375 (5)	0.0389 (5)	−0.0009 (4)	−0.0177 (4)	−0.0046 (4)
C14	0.0865 (11)	0.0433 (7)	0.0834 (11)	0.0160 (7)	−0.0373 (9)	0.0012 (7)
C15	0.0398 (5)	0.0351 (5)	0.0400 (6)	−0.0048 (4)	−0.0158 (4)	0.0010 (4)
C16	0.0561 (7)	0.0544 (7)	0.0385 (6)	−0.0177 (6)	−0.0094 (5)	0.0022 (5)
C17	0.0555 (7)	0.0417 (6)	0.0492 (7)	−0.0115 (5)	−0.0243 (6)	0.0056 (5)
C18	0.0771 (10)	0.0371 (6)	0.0793 (10)	−0.0093 (6)	−0.0426 (8)	0.0029 (6)
C19	0.0688 (9)	0.0489 (7)	0.0816 (11)	0.0077 (6)	−0.0408 (8)	−0.0205 (7)
C20	0.0486 (7)	0.0646 (8)	0.0584 (8)	0.0025 (6)	−0.0205 (6)	−0.0209 (7)
C21	0.0416 (6)	0.0511 (7)	0.0463 (6)	−0.0083 (5)	−0.0150 (5)	−0.0063 (5)
C22	0.0391 (5)	0.0378 (5)	0.0428 (6)	−0.0057 (4)	−0.0184 (5)	−0.0020 (4)
C23	0.0426 (5)	0.0341 (5)	0.0305 (5)	−0.0078 (4)	−0.0121 (4)	0.0003 (4)
C24	0.0506 (6)	0.0419 (6)	0.0395 (6)	0.0020 (5)	−0.0155 (5)	−0.0075 (5)
C25	0.0706 (9)	0.0530 (7)	0.0369 (6)	0.0040 (6)	−0.0159 (6)	−0.0139 (5)
C26	0.0725 (8)	0.0458 (6)	0.0394 (6)	−0.0056 (6)	−0.0270 (6)	−0.0042 (5)
C27	0.0556 (7)	0.0434 (6)	0.0462 (6)	−0.0016 (5)	−0.0243 (6)	0.0003 (5)
C28	0.0449 (6)	0.0432 (6)	0.0368 (6)	−0.0017 (5)	−0.0115 (5)	−0.0019 (5)
C29	0.1203 (16)	0.0809 (11)	0.0495 (8)	0.0099 (11)	−0.0479 (10)	−0.0122 (8)

Geometric parameters (Å, º)

O1—C12	1.2271 (16)	C9—C10	1.389 (2)
O2—C13	1.2019 (14)	C9—H9	0.9300
O3—C13	1.3437 (14)	C10—C11	1.3888 (18)
O3—C14	1.4492 (16)	C10—H10	0.9300
N1—C5	1.4662 (16)	C14—H14A	0.9600
N1—C1	1.4668 (16)	C14—H14B	0.9600
N1—C4	1.4732 (14)	C14—H14C	0.9600
N2—C12	1.3555 (16)	C15—H15A	0.9700
N2—C11	1.4055 (17)	C15—H15B	0.9700
N2—H2	0.8600	C16—H16	0.9300
N3—C16	1.3657 (15)	C17—C22	1.4065 (16)
N3—C22	1.4000 (16)	C17—C18	1.409 (2)
N3—C15	1.4639 (14)	C18—C19	1.376 (2)
N4—C16	1.3172 (19)	C18—H18	0.9300

N4—C17	1.3981 (19)	C19—C20	1.400 (2)
C1—C2	1.5467 (16)	C19—H19	0.9300
C1—H1A	0.9700	C20—C21	1.3866 (19)
C1—H1B	0.9700	C20—H20	0.9300
C2—C23	1.5247 (14)	C21—C22	1.3973 (17)
C2—C3	1.5921 (14)	C21—H21	0.9300
C2—H2A	0.9800	C23—C24	1.3968 (16)
C3—C13	1.5357 (15)	C23—C28	1.3998 (16)
C3—C15	1.5735 (15)	C24—C25	1.3976 (18)
C3—C4	1.5840 (15)	C24—H24	0.9300
C4—C6	1.5396 (16)	C25—C26	1.393 (2)
C4—C12	1.5658 (15)	C25—H25	0.9300
C5—H5A	0.9600	C26—C27	1.391 (2)
C5—H5B	0.9600	C26—C29	1.5173 (18)
C5—H5C	0.9600	C27—C28	1.3934 (17)
C6—C7	1.3915 (17)	C27—H27	0.9300
C6—C11	1.4021 (16)	C28—H28	0.9300
C7—C8	1.4033 (18)	C29—H29A	0.9600
C7—H7	0.9300	C29—H29B	0.9600
C8—C9	1.386 (2)	C29—H29C	0.9600
C8—H8	0.9300		

C13—O3—C14	115.61 (11)	N2—C12—C4	108.20 (10)
C5—N1—C1	113.09 (10)	O2—C13—O3	124.23 (10)
C5—N1—C4	115.51 (9)	O2—C13—C3	124.75 (10)
C1—N1—C4	106.21 (9)	O3—C13—C3	111.01 (9)
C12—N2—C11	112.18 (10)	O3—C14—H14A	109.5
C12—N2—H2	123.9	O3—C14—H14B	109.5
C11—N2—H2	123.9	H14A—C14—H14B	109.5
C16—N3—C22	105.96 (10)	O3—C14—H14C	109.5
C16—N3—C15	128.19 (11)	H14A—C14—H14C	109.5
C22—N3—C15	125.72 (9)	H14B—C14—H14C	109.5
C16—N4—C17	104.88 (11)	N3—C15—C3	116.04 (9)
N1—C1—C2	105.55 (9)	N3—C15—H15A	108.3
N1—C1—H1A	110.6	C3—C15—H15A	108.3
C2—C1—H1A	110.6	N3—C15—H15B	108.3
N1—C1—H1B	110.6	C3—C15—H15B	108.3
C2—C1—H1B	110.6	H15A—C15—H15B	107.4
H1A—C1—H1B	108.8	N4—C16—N3	114.03 (12)
C23—C2—C1	114.32 (9)	N4—C16—H16	123.0
C23—C2—C3	117.51 (9)	N3—C16—H16	123.0
C1—C2—C3	104.77 (8)	N4—C17—C22	109.58 (11)
C23—C2—H2A	106.5	N4—C17—C18	130.71 (12)
C1—C2—H2A	106.5	C22—C17—C18	119.71 (13)
C3—C2—H2A	106.5	C19—C18—C17	117.84 (13)
C13—C3—C15	109.64 (9)	C19—C18—H18	121.1
C13—C3—C4	112.06 (8)	C17—C18—H18	121.1
C15—C3—C4	113.01 (9)	C18—C19—C20	121.77 (14)

C13—C3—C2	109.12 (9)	C18—C19—H19	119.1
C15—C3—C2	112.02 (8)	C20—C19—H19	119.1
C4—C3—C2	100.71 (8)	C21—C20—C19	121.69 (14)
N1—C4—C6	118.20 (9)	C21—C20—H20	119.2
N1—C4—C12	108.16 (9)	C19—C20—H20	119.2
C6—C4—C12	101.17 (9)	C20—C21—C22	116.65 (12)
N1—C4—C3	100.65 (8)	C20—C21—H21	121.7
C6—C4—C3	112.70 (9)	C22—C21—H21	121.7
C12—C4—C3	116.68 (9)	C21—C22—N3	132.09 (11)
N1—C5—H5A	109.5	C21—C22—C17	122.34 (12)
N1—C5—H5B	109.5	N3—C22—C17	105.55 (10)
H5A—C5—H5B	109.5	C24—C23—C28	117.53 (10)
N1—C5—H5C	109.5	C24—C23—C2	120.08 (10)
H5A—C5—H5C	109.5	C28—C23—C2	122.35 (10)
H5B—C5—H5C	109.5	C23—C24—C25	120.96 (12)
C7—C6—C11	118.67 (11)	C23—C24—H24	119.5
C7—C6—C4	133.14 (10)	C25—C24—H24	119.5
C11—C6—C4	108.15 (10)	C26—C25—C24	121.19 (12)
C6—C7—C8	119.29 (12)	C26—C25—H25	119.4
C6—C7—H7	120.4	C24—C25—H25	119.4
C8—C7—H7	120.4	C27—C26—C25	118.01 (11)
C9—C8—C7	120.61 (13)	C27—C26—C29	120.20 (14)
C9—C8—H8	119.7	C25—C26—C29	121.79 (14)
C7—C8—H8	119.7	C26—C27—C28	120.98 (12)
C8—C9—C10	121.15 (13)	C26—C27—H27	119.5
C8—C9—H9	119.4	C28—C27—H27	119.5
C10—C9—H9	119.4	C27—C28—C23	121.32 (11)
C9—C10—C11	117.67 (13)	C27—C28—H28	119.3
C9—C10—H10	121.2	C23—C28—H28	119.3
C11—C10—H10	121.2	C26—C29—H29A	109.5
C10—C11—C6	122.60 (13)	C26—C29—H29B	109.5
C10—C11—N2	127.34 (12)	H29A—C29—H29B	109.5
C6—C11—N2	110.06 (10)	C26—C29—H29C	109.5
O1—C12—N2	126.27 (11)	H29A—C29—H29C	109.5
O1—C12—C4	125.45 (11)	H29B—C29—H29C	109.5

C5—N1—C1—C2	161.69 (10)	C6—C4—C12—N2	−4.58 (12)
C4—N1—C1—C2	33.99 (12)	C3—C4—C12—N2	118.07 (11)
N1—C1—C2—C23	124.14 (10)	C14—O3—C13—O2	−2.91 (18)
N1—C1—C2—C3	−5.90 (12)	C14—O3—C13—C3	176.04 (12)
C23—C2—C3—C13	92.40 (11)	C15—C3—C13—O2	104.57 (13)
C1—C2—C3—C13	−139.46 (9)	C4—C3—C13—O2	−129.11 (12)
C23—C2—C3—C15	−29.19 (13)	C2—C3—C13—O2	−18.46 (15)
C1—C2—C3—C15	98.95 (10)	C15—C3—C13—O3	−74.38 (11)
C23—C2—C3—C4	−149.56 (10)	C4—C3—C13—O3	51.94 (12)
C1—C2—C3—C4	−21.42 (11)	C2—C3—C13—O3	162.60 (9)
C5—N1—C4—C6	−50.74 (14)	C16—N3—C15—C3	92.19 (15)
C1—N1—C4—C6	75.51 (12)	C22—N3—C15—C3	−83.07 (13)

C5—N1—C4—C12	63.29 (13)	C13—C3—C15—N3	26.13 (13)
C1—N1—C4—C12	−170.47 (9)	C4—C3—C15—N3	−99.65 (11)
C5—N1—C4—C3	−173.86 (10)	C2—C3—C15—N3	147.43 (9)
C1—N1—C4—C3	−47.62 (11)	C17—N4—C16—N3	−0.14 (17)
C13—C3—C4—N1	156.94 (9)	C22—N3—C16—N4	0.01 (16)
C15—C3—C4—N1	−78.59 (10)	C15—N3—C16—N4	−175.99 (12)
C2—C3—C4—N1	41.06 (10)	C16—N4—C17—C22	0.22 (15)
C13—C3—C4—C6	30.07 (12)	C16—N4—C17—C18	−178.59 (15)
C15—C3—C4—C6	154.54 (9)	N4—C17—C18—C19	178.97 (14)
C2—C3—C4—C6	−85.80 (10)	C22—C17—C18—C19	0.3 (2)
C13—C3—C4—C12	−86.36 (12)	C17—C18—C19—C20	−0.2 (2)
C15—C3—C4—C12	38.11 (13)	C18—C19—C20—C21	0.0 (2)
C2—C3—C4—C12	157.77 (9)	C19—C20—C21—C22	0.17 (19)
N1—C4—C6—C7	−59.69 (17)	C20—C21—C22—N3	−178.63 (12)
C12—C4—C6—C7	−177.48 (13)	C20—C21—C22—C17	−0.07 (18)
C3—C4—C6—C7	57.16 (16)	C16—N3—C22—C21	178.86 (13)
N1—C4—C6—C11	122.37 (11)	C15—N3—C22—C21	−5.02 (19)
C12—C4—C6—C11	4.58 (11)	C16—N3—C22—C17	0.13 (13)
C3—C4—C6—C11	−120.78 (10)	C15—N3—C22—C17	176.25 (10)
C11—C6—C7—C8	0.58 (18)	N4—C17—C22—C21	−179.10 (11)
C4—C6—C7—C8	−177.19 (12)	C18—C17—C22—C21	−0.14 (19)
C6—C7—C8—C9	0.4 (2)	N4—C17—C22—N3	−0.21 (14)
C7—C8—C9—C10	−0.6 (2)	C18—C17—C22—N3	178.75 (11)
C8—C9—C10—C11	−0.1 (2)	C1—C2—C23—C24	139.36 (11)
C9—C10—C11—C6	1.2 (2)	C3—C2—C23—C24	−97.22 (12)
C9—C10—C11—N2	−178.74 (13)	C1—C2—C23—C28	−38.27 (14)
C7—C6—C11—C10	−1.40 (19)	C3—C2—C23—C28	85.16 (13)
C4—C6—C11—C10	176.88 (12)	C28—C23—C24—C25	0.84 (18)
C7—C6—C11—N2	178.53 (11)	C2—C23—C24—C25	−176.90 (11)
C4—C6—C11—N2	−3.18 (13)	C23—C24—C25—C26	−0.3 (2)
C12—N2—C11—C10	179.99 (13)	C24—C25—C26—C27	−1.1 (2)
C12—N2—C11—C6	0.05 (15)	C24—C25—C26—C29	178.44 (15)
C11—N2—C12—O1	−173.75 (12)	C25—C26—C27—C28	2.0 (2)
C11—N2—C12—C4	3.05 (14)	C29—C26—C27—C28	−177.59 (14)
N1—C4—C12—O1	47.38 (15)	C26—C27—C28—C23	−1.45 (19)
C6—C4—C12—O1	172.25 (12)	C24—C23—C28—C27	0.02 (17)
C3—C4—C12—O1	−65.10 (16)	C2—C23—C28—C27	177.70 (10)
N1—C4—C12—N2	−129.44 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N4^{i}	0.86	2.05	2.888 (2)	165
C16—H16···O1	0.93	2.33	3.050 (2)	134

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