Methyl 1-ethyl-3-[hydroxy(naphthalen-1-yl)methyl]-1-methyl-2-oxospiro[indoline-3,2-pyrrolidine]-3-carboxylate

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Methyl 1-ethyl-3’-[hydroxy(naphthalen-1-yl)methyl]-1’-methyl-2-oxospiro[indoline-3,2’-pyrrolidine]-3’-carboxylate

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Key indicators: single-crystal X-ray study; T = 293 K; mean [C–C] = 0.002 Å; R factor = 0.039; wR factor = 0.111; data-to-parameter ratio = 15.5.

In the title compound, C27H28N2O4, the pyrrolidine ring adopts a twist conformation. The plane of the indole ring is almost perpendicular to that of the pyrrolidine ring, making a dihedral angle of 88.50 (6)°. The planes of the naphthyl ring system and the pyrrolidine ring are tilted by an angle of 55.86 (5)°. The molecular conformation is stabilized by intramolecular O—H⋯O and O—H⋯N hydrogen bonds.

Related literature
For general background to spiro compounds and their biological activity, see: Pradhan et al. (2006); For uses of pyrrolidine derivative, see: Amal Raj et al. (2003); For conformation studies, see: Nardelli (1983).

Experimental

Crystal data

C27H28N2O4

M = 444.51

Orthorhombic, Pbca

V = 4547.23 (16) Å³

Z = 8

Mo Kα radiation

μ = 0.09 mm⁻¹

T = 293 K

Tmin = 0.979, Tmax = 0.983

data collection

Bruker Kappa APEXII CCD diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2004)

Tmin = 0.25, Tmax = 0.20

I > 2σ(I)

Rint = 0.041

Refinement

R[F² > 2σ(F²)] = 0.039

wR(F²) = 0.111

S = 1.02

4636 reflections

299 parameters

H-atom parameters constrained

Δρmax = 0.02 e Å⁻³

Δρmin = −0.14 e Å⁻³

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

D—H⋯Å

D—H

H⋯Å

D⋯Å

D—H⋯Å

O1—H1⋯O4

0.82

2.37

2.9121 (16)

124

O1—H1⋯N1

0.82

2.39

2.9439 (17)

126

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2 and SAINT (Bruker, 2004); data reduction: SAINT and XPREP (Bruker, 2004); 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, 2012); software used to prepare material for publication: PLATON (Spek, 2009).

Supporting information for this paper is available from the IUCr electronic archives (Reference: BT6950).

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supplementary materials

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Methyl 1-ethyl-3’-[hydroxy(naphthalen-1-yl)methyl]-1’-methyl-2-oxospiro-
[indoline-3,2’-pyrrolidine]-3’-carboxylate

Vinodhkumar Vijayakumar, Gunther H. Peters, M. Suresh, Raghunathan Raghavachary and G. Jagadeesan

1. Comment
Spiro compounds have received considerable interest due to their biological properties (Pradhan et al., 2006). In addition, pyrrolidine derivatives are found to have anticonvulsant, antimicrobial and antifungal activities against various pathogens (Amal Raj et al., 2003). In view of their importance, the crystal structure determination of the title compound was carried out and the results are presented herein. In the title molecule (Fig. 1) the five-membered pyrrolidine ring [DS (N1) = 0.101 (1) Å and D2 (C10) = 0.051 (9) Å] adopts a twist conformation defined by the above asymmetry parameters (Nardelli, 1983). The indole ring (C1—C8/N2) is almost perpendicular to the pyrrolidine ring with dihedral angle of 88.50 (6)°. The naphthyl and pyrrolidine rings are tilted by an angle of 55.86 (5)°. The molecular conformation is stabilized by an intramolecular O—H···O and O—H···N hydrogen bond (Fig. 2 and Table 1).

2. Experimental
A mixture of methyl 2-(hydroxy(naphthalen-1-yl)methyl)acrylate (1 mmol), N-ethyl isatin (1.1 mmol) and sarcosine (1.1 mmol) was refluxed in methanol until completion of the reaction was evidenced by TLC analysis. After completion of the reaction the solvent was evaporated under reduced pressure. The reaction mixture was dissolved in ethyl acetate and washed with water followed by brine solution. The organic layer was separated and evaporated under reduced pressure. The crude mixture was purified by column chromatography using ethyl acetate and hexane as eluent (3: 7). The product was dissolved in ethyl acetate and heated for two minutes. The resulting solution was subjected to crystallization by slow evaporation of the solvent for 48 h resulting in the formation of single crystals.

3. Refinement
All H atoms were positioned geometrically, with C–H = 0.93–0.97 Å and constrained to ride on their parent atom with $U_{iso}(H) = 1.5U_{eq}(O,C)$ for methyl H atoms and $1.2U_{eq}(C)$ for other H atoms.
Figure 1
The molecular structure of the title compound, Displacement ellipsoids are drawn at the 30% probability level, H atoms have been omitted for clarity.
Figure 2
Crystal packing of the title compound, Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the interactions have been omitted.

(I)

Crystal data

*C*<sub>2</sub>*H*<sub>3</sub>*N*<sub>2</sub>*O*<sub>4</sub>  
*M*<sub>r</sub> = 444.51  
Orthorhombic, *Pbca*  
Hall symbol: -*P 2ac 2ab*  
*a* = 16.7802 (3) Å  
*b* = 14.6690 (3) Å  
*c* = 18.4735 (4) Å  
*V* = 4547.23 (16) Å<sup>3</sup>  
*Z* = 8

F(000) = 1888  
*D*<sub>c</sub> = 1.299 Mg m<sup>-3</sup>  
Mo *Ka* radiation, *λ* = 0.71073 Å  
Cell parameters from 8834 reflections  
*θ* = 2.1–31.2°  
*μ* = 0.09 mm<sup>-1</sup>  
*T* = 293 K  
Block, colourless  
0.25 × 0.20 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
ω and φ scan  
Absorption correction: multi-scan  
(SADABS; Bruker, 2004)  
*R*<sub>int</sub> = 0.041  
θ<sub>max</sub> = 26.4°, θ<sub>min</sub> = 2.2°  
44640 measured reflections  
4636 independent reflections  
3429 reflections with *I* > 2σ(*I*)  
h = −20→18  
k = −18→18  
l = −23→23

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**Refinement**

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.111$

$S = 1.02$

4636 reflections

299 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

$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 1.1973P]$ where $P = (P_o^2 + 2P_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.22$ e Å$^{-3}$

$\Delta\rho_{\text{min}} = -0.14$ e Å$^{-3}$

Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^*=kF_c[1+0.001xF_c^2/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0061 (4)

**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 on $F^2$ 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$^2$)**

| Atom | x    | y    | z    | $U_{	ext{iso}}$/$U_{	ext{eq}}$ |
|------|------|------|------|-----------------------------|
| O1   | 0.96460 (6) | 0.22318 (7) | 0.34518 (6) | 0.0455 (3) |
| H1   | 0.9670  | 0.1849 | 0.3775 | 0.068* |
| O3   | 0.90834 (6) | 0.42866 (7) | 0.49176 (5) | 0.0417 (3) |
| O2   | 0.78729 (7) | 0.43761 (8) | 0.44130 (6) | 0.0539 (3) |
| N1   | 0.86301 (8) | 0.14862 (9) | 0.46083 (7) | 0.0430 (3) |
| O4   | 1.02618 (7) | 0.21025 (9) | 0.49220 (6) | 0.0573 (3) |
| N2   | 0.96210 (8) | 0.25963 (9) | 0.59423 (7) | 0.0456 (3) |
| C18  | 1.01590 (8) | 0.40195 (9) | 0.28012 (7) | 0.0332 (3) |
| C13  | 0.94194 (8) | 0.37632 (10) | 0.31325 (7) | 0.0328 (3) |
| C12  | 0.94494 (8) | 0.30940 (9) | 0.37557 (7) | 0.0332 (3) |
| H12  | 0.9891  | 0.3276 | 0.4071 | 0.040* |
| C17  | 1.01480 (9) | 0.46284 (10) | 0.22021 (8) | 0.0388 (3) |
| C3   | 0.88393 (10) | 0.28059 (10) | 0.61479 (8) | 0.0432 (4) |
| C1   | 0.88143 (9) | 0.23837 (10) | 0.49136 (7) | 0.0360 (3) |
| C4   | 0.83254 (10) | 0.26839 (10) | 0.55663 (8) | 0.0397 (4) |
| C10  | 0.79905 (9) | 0.25417 (11) | 0.38403 (8) | 0.0410 (4) |
| H10A | 0.7503  | 0.2582 | 0.3892 | 0.049* |
| H10B | 0.8108  | 0.2476 | 0.3329 | 0.049* |
| C19  | 1.09071 (9) | 0.36786 (11) | 0.30294 (8) | 0.0395 (4) |
| H19  | 1.0931  | 0.3281 | 0.3421 | 0.047* |
| C11  | 0.88684 (8) | 0.30242 (10) | 0.42346 (7) | 0.0333 (3) |
| C23  | 0.84805 (9) | 0.39681 (10) | 0.45129 (7) | 0.0364 (3) |
| C16  | 0.94110 (10) | 0.49640 (11) | 0.19414 (9) | 0.0459 (4) |
| H16  | 0.9401  | 0.5363 | 0.1550 | 0.055* |
### Atomic displacement parameters (Å²)

|          | U₁₁  | U₂₂  | U₃₃  | U₁₂  | U₁₃  | U₂₃  |
|----------|------|------|------|------|------|------|
| O1       | 0.0565 (7) | 0.0361 (6) | 0.0438 (6) | 0.0055 (5) | 0.0104 (5) | −0.0025 (5) |
| O3       | 0.0426 (6) | 0.0372 (6) | 0.0452 (6) | −0.0019 (4) | −0.0030 (5) | −0.0087 (5) |
| O2       | 0.0459 (7) | 0.0586 (7) | 0.0571 (7) | 0.0166 (5) | −0.0053 (5) | −0.0069 (6) |
| N1       | 0.0528 (8) | 0.0357 (7) | 0.0404 (7) | −0.0048 (6) | −0.0031 (6) | −0.0018 (6) |
| O4       | 0.0428 (7) | 0.0746 (9) | 0.0544 (7) | 0.0114 (6) | −0.0006 (6) | 0.0086 (6) |
| N2       | 0.0503 (8) | 0.0489 (8) | 0.0377 (7) | −0.0043 (6) | −0.0115 (6) | 0.0051 (6) |
| C18      | 0.0329 (7) | 0.0321 (7) | 0.0347 (7) | −0.0002 (6) | 0.0017 (6) | −0.0043 (6) |
| C13      | 0.0315 (8) | 0.0346 (8) | 0.0324 (7) | −0.0005 (6) | −0.0002 (6) | −0.0042 (6) |
| C12      | 0.0311 (7) | 0.0341 (8) | 0.0343 (7) | −0.0009 (6) | 0.0005 (6) | −0.0036 (6) |

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*Acta Cryst. (2014). E70, o540*
### Geometric parameters (Å, °)

|          |          |          |          |          |          |
|----------|----------|----------|----------|----------|----------|
| O1—C12  | 1.4224 (17) | C11—C23 | 1.518 (2) |          |          |
| O1—H1   | 0.8200    | C16—C15 | 1.353 (2) |          |          |
| O3—C23  | 1.3418 (17) | C16—H16 | 0.9300    |          |          |
| O3—C24  | 1.4392 (19) | C14—C15 | 1.401 (2) |          |          |
| O2—C2   | 1.1965 (17) | C14—H14 | 0.9300    |          |          |
| N1—C27  | 1.457 (2)   | C20—C21 | 1.399 (2) |          |          |
| N1—C9   | 1.458 (2)   | C20—H20 | 0.9300    |          |          |
| N1—C1   | 1.4652 (19) | C9—H9A  | 0.9700    |          |          |
| O4—C2   | 1.2185 (19) | C9—H9B  | 0.9700    |          |          |
| N2—C2   | 1.355 (2)   | C8—C7   | 1.382 (3) |          |          |
| N2—C3   | 1.400 (2)   | C8—H8   | 0.9300    |          |          |
| N2—C25  | 1.456 (2)   | C21—C22 | 1.355 (2) |          |          |
| C18—C19 | 1.415 (2)   | C21—H21 | 0.9300    |          |          |
| C18—C17 | 1.422 (2)   | C5—C6   | 1.391 (2) |          |          |
| C18—C13 | 1.4339 (19) | C5—H5   | 0.9300    |          |          |
| C13—C14 | 1.368 (2)   | C22—H22 | 0.9300    |          |          |
| C13—C12 | 1.514 (2)   | C27—H27A | 0.9600  |          |          |
| C12—C11 | 1.5570 (19) | C27—H27B | 0.9600  |          |          |
| C12—H12 | 0.9800     | C27—H27C | 0.9600  |          |          |
| C17—C22 | 1.410 (2)   | C24—H24A | 0.9600  |          |          |
| C17—C16 | 1.416 (2)   | C24—H24B | 0.9600  |          |          |
| C3—C8   | 1.380 (2)   | C24—H24C | 0.9600  |          |          |
| C3—C4   | 1.389 (2)   | C6—C7   | 1.368 (3) |          |          |
| Bond                  | Distance (Å) | Bond                  | Distance (Å) | Dist. (%) |
|----------------------|--------------|----------------------|--------------|-----------|
| C1—C4                | 1.523 (2)    | C6—H6                | 0.930        |
| C1—C2                | 1.542 (2)    | C25—C26              | 1.490 (3)    |
| C1—C11               | 1.581 (2)    | C25—H25A             | 0.970        |
| C4—C5                | 1.378 (2)    | C25—H25B             | 0.970        |
| C10—C9               | 1.519 (2)    | C15—H15              | 0.930        |
| C10—C11              | 1.5501 (19)  | C7—H7                | 0.930        |
| C10—H10A             | 0.9700       | C26—H26A             | 0.960        |
| C10—H10B             | 0.9700       | C26—H26B             | 0.960        |
| C19—C20              | 1.358 (2)    | C26—H26C             | 0.960        |
| C19—H19              | 0.9300       |                      |              |
| C12—O1—H1            | 109.5        | C15—C14—H14          | 118.9        |
| C23—O3—C24           | 116.77 (12)  | C19—C20—C21          | 120.76 (15)  |
| C27—N1—C9            | 114.27 (13)  | C19—C20—H20          | 119.6        |
| C27—N1—C1            | 115.31 (12)  | C21—C20—H20          | 119.6        |
| C9—N1—C1             | 105.46 (12)  | O4—C2—N2             | 125.39 (15)  |
| C2—N2—C3             | 111.49 (13)  | O4—C2—C1             | 126.01 (14)  |
| C2—N2—C25            | 123.11 (15)  | N2—C2—C1             | 108.53 (13)  |
| C3—N2—C25            | 125.40 (14)  | N1—C9—C10            | 104.79 (12)  |
| C19—C18—C17          | 117.71 (13)  | N1—C9—H9A            | 110.8        |
| C19—C18—C13          | 123.22 (13)  | C10—C9—H9A           | 110.8        |
| C17—C18—C13          | 119.05 (13)  | N1—C9—H9B            | 110.8        |
| C14—C13—C18          | 118.43 (13)  | C10—C9—H9B           | 110.8        |
| C14—C13—C12          | 123.76 (13)  | H9A—C9—H9B           | 108.9        |
| C18—C13—C12          | 117.77 (12)  | C3—C8—C7             | 117.37 (17)  |
| O1—C12—C13           | 106.51 (11)  | C3—C8—H8             | 121.3        |
| O1—C12—C11           | 110.86 (11)  | C7—C8—H8             | 121.3        |
| C13—C12—C11          | 116.59 (11)  | C22—C21—C20          | 120.08 (15)  |
| O1—C12—H12           | 107.5        | C22—C21—H21          | 120.0        |
| C13—C12—H12          | 107.5        | C20—C21—H21          | 120.0        |
| C11—C12—H12          | 107.5        | C4—C5—C6             | 118.98 (17)  |
| C22—C17—C16          | 120.97 (14)  | C4—C5—H5             | 120.5        |
| C22—C17—C18          | 119.38 (14)  | C6—C5—H5             | 120.5        |
| C16—C17—C18          | 119.64 (13)  | C21—C22—C17          | 120.94 (15)  |
| C8—C3—C4             | 122.11 (17)  | C21—C22—H22          | 119.5        |
| C8—C3—N2             | 127.79 (16)  | C17—C22—H22          | 119.5        |
| C4—C3—N2             | 110.09 (13)  | N1—C27—H27A          | 109.5        |
| N1—C1—C4             | 116.80 (12)  | N1—C27—H27B          | 109.5        |
| N1—C1—C2             | 108.27 (12)  | H27A—C27—H27B        | 109.5        |
| C4—C1—C2             | 101.51 (12)  | N1—C27—H27C          | 109.5        |
| N1—C1—C11            | 101.56 (11)  | H27A—C27—H27C        | 109.5        |
| C4—C1—C11            | 112.59 (12)  | H27B—C27—H27C        | 109.5        |
| C2—C1—C11            | 116.72 (12)  | O3—C24—H24A          | 109.5        |
| C5—C4—C3             | 119.39 (14)  | O3—C24—H24B          | 109.5        |
| C5—C4—C1             | 132.19 (14)  | H24A—C24—H24B        | 109.5        |
| C3—C4—C1             | 108.37 (13)  | O3—C24—H24C          | 109.5        |
| C9—C10—C11           | 106.51 (12)  | H24A—C24—H24C        | 109.5        |
| C9—C10—H10A          | 110.4        | H24B—C24—H24C        | 109.5        |
| C11—C10—H10A         | 110.4        | C7—C6—C5             | 120.56 (18)  |
C9—C10—H10B 110.4  C7—C6—H6 119.7
C11—C10—H10B 110.4  C5—C6—H6 119.7
H10A—C10—H10B 110.4  N2—C25—C26 113.18 (16)
C20—C19—C18 121.12 (14)  N2—C25—H25A 108.9
C20—C19—H19 119.4  C26—C25—H25A 108.9
C18—C19—H19 119.4  C26—C25—H25B 108.9
C23—C11—C10 113.72 (12)  C26—C25—H25B 107.8
C23—C11—C12 108.73 (11)  C16—C15—C14 120.62 (15)
C10—C11—C12 112.50 (11)  C16—C15—H15 119.7
C23—C11—C1 107.70 (11)  C14—C15—H15 119.7
C10—C11—C1 101.67 (11)  C6—C7—C8 121.59 (17)
C12—C11—C1 112.35 (11)  C6—C7—H7 119.2
O2—C23—O3 123.65 (14)  C8—C7—H7 119.2
O2—C23—C11 126.86 (14)  C25—C26—H26A 109.5
O3—C23—C11 109.47 (12)  C25—C26—H26B 109.5
C15—C16—C17 120.09 (14)  C25—C26—H26C 109.5
C15—C16—C16H6 120.0  H26A—C26—H26B 109.5
C17—C16—C16H6 120.0  H26A—C26—H26C 109.5
C13—C14—C15 122.18 (14)  H26B—C26—H26C 109.5
C13—C14—H14 118.9  

C19—C18—C13—C14 −177.76 (14)  C4—C1—C11—C10 −91.23 (14)
C17—C18—C13—C14 0.7 (2)  C2—C1—C11—C10 151.93 (12)
C19—C18—C13—C12 −0.1 (2)  N1—C1—C11—C10 −86.02 (13)
C17—C18—C13—C12 178.35 (12)  C4—C1—C11—C12 148.28 (12)
C14—C13—C12—O1 106.00 (15)  C2—C1—C11—C12 −91.23 (14)
C18—C13—C12—O1 −71.55 (15)  C24—O3—C23—O2 12.4 (2)
C14—C13—C12—C11 183.3 (2)  C24—O3—C23—C11 −165.93 (13)
C18—C13—C12—C11 183.3 (2)  C10—C11—C23—C11 −172.73 (11)
C19—C18—C17—C22 −0.3 (2)  C12—C11—C23—O2 −5.6 (2)
C13—C18—C17—C22 −178.76 (14)  C25—C25—C26—H26A 109.5
C19—C18—C17—C16 −178.06 (14)  C25—C25—C26—H26B 109.5
C13—C18—C17—C16 −0.4 (2)  C25—C25—C26—H26C 109.5
C2—N2—C3—C4 −177.79 (16)  C10—C11—C23—O3 172.73 (11)
C25—N2—C3—C4 3.1 (3)  C12—C11—C23—O3 −61.09 (14)
C2—N2—C3—C8 0.98 (18)  C11—C11—C23—O2 −117.41 (16)
C25—N2—C3—C8 −178.11 (14)  C10—C11—C23—O2 172.73 (11)
C27—N1—C1—C4 77.10 (15)  C12—C11—C23—O3 −177.87 (14)
C27—N1—C1—C14 49.96 (19)  C18—C19—C29—C21 0.3 (3)
C9—N1—C1—C4 77.10 (15)  C3—N2—C2—O4 −177.41 (15)
C9—N1—C1—C14 63.76 (17)  C25—N2—C2—O4 −177.87 (14)
C27—N1—C1—C2 −169.18 (12)  C3—N2—C2—O4 1.7 (3)
C27—N1—C1—C11 −172.81 (14)  C3—N2—C2—C1 0.3 (3)
C9—N1—C1—C11 −45.76 (14)  C25—N2—C2—C1 0.3 (3)
C8—C3—C4—C5 0.2 (2)  C25—N2—C2—C1 −178.75 (14)
N2—C3—C4—C5 −178.66 (14)  N1—C1—C2—O4 53.2 (2)
C8—C3—C4—C1 177.69 (15)  C4—C1—C2—O4 176.71 (16)
N2—C3—C4—C1 −1.16 (17)  C11—C1—C2—O4 −60.5 (2)
N1—C1—C4—C5 −64.6 (2)  N1—C1—C2—N2 −123.80 (13)
C2—C1—C4—C5 177.95 (17)  C4—C1—C2—N2 −0.32 (15)
C11—C1—C2—N2 122.47 (13)
supplementary materials

C11—C1—C4—C5 52.4 (2) C27—N1—C9—C10 165.99 (14)
N1—C1—C4—C3 118.35 (14) C1—N1—C9—C10 38.31 (15)
C2—C1—C4—C3 0.88 (15) C11—C10—C9—N1 −14.51 (16)
C11—C1—C4—C3 −124.70 (13) C4—C3—C8—C7 0.0 (3)
C17—C18—C19—C20 −0.2 (2) N2—C3—C8—C7 178.67 (16)
C13—C18—C19—C20 178.25 (15) C19—C20—C21—C22 0.0 (3)
C9—C10—C11—C23 −127.72 (13) C3—C4—C5—C6 −0.3 (2)
C9—C10—C11—C12 108.12 (13) C1—C4—C5—C6 −177.12 (16)
C9—C10—C11—C1 12.26 (15) C20—C21—C22—C17 −0.4 (3)
O1—C12—C11—C23 −177.09 (11) C13—C12—C11—C10 −55.04 (15)
C13—C12—C11—C10 50.22 (15) C18—C17—C22—C21 0.6 (3)
C13—C12—C11—C1 −174.14 (11) C4—C5—C6—C7 0.2 (3)
N1—C1—C11—C23 154.27 (11) C17—C16—C15—C14 0.5 (3)
N1—C1—C11—C10 34.47 (13) C13—C14—C15—C16 −0.2 (3)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O1—H1···O4 | 0.82 | 2.37 | 2.9121 (16) | 124 |
| O1—H1···N1 | 0.82 | 2.39 | 2.9439 (17) | 126 |