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## Structure Reports

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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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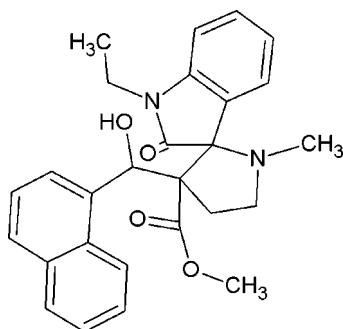
Received 12 December 2013; accepted 30 March 2014

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.111; data-to-parameter ratio = 15.5.

In the title compound,  $\text{C}_{27}\text{H}_{28}\text{N}_2\text{O}_4$ , 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  $88.50(6)^\circ$ . The planes of the naphthyl ring system and the pyrrolidine ring are tilted by an angle of  $55.86(5)^\circ$ . The molecular conformation is stabilized by intramolecular  $\text{O}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{N}$  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

$\text{C}_{27}\text{H}_{28}\text{N}_2\text{O}_4$   
 $M_r = 444.51$   
 Orthorhombic,  $Pbca$   
 $a = 16.7802(3)$  Å  
 $b = 14.6690(3)$  Å  
 $c = 18.4735(4)$  Å  
 $V = 4547.23(16)$  Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.25 \times 0.20 \times 0.20$  mm

## Data collection

Bruker Kappa APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2004)  
 $T_{\min} = 0.979$ ,  $T_{\max} = 0.983$   
 44640 measured reflections  
 4636 independent reflections  
 3429 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.041$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.111$   
 $S = 1.02$   
 4636 reflections  
 299 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.22$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.14$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                        | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------------------------|-------|-------------|-------------|---------------|
| $\text{O1}-\text{H1}\cdots\text{O4}$ | 0.82  | 2.37        | 2.9121 (16) | 124           |
| $\text{O1}-\text{H1}\cdots\text{N1}$ | 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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## supporting information

*Acta Cryst.* (2014). E70, o540 [doi:10.1107/S1600536814007065]

## 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

### S1. Comment

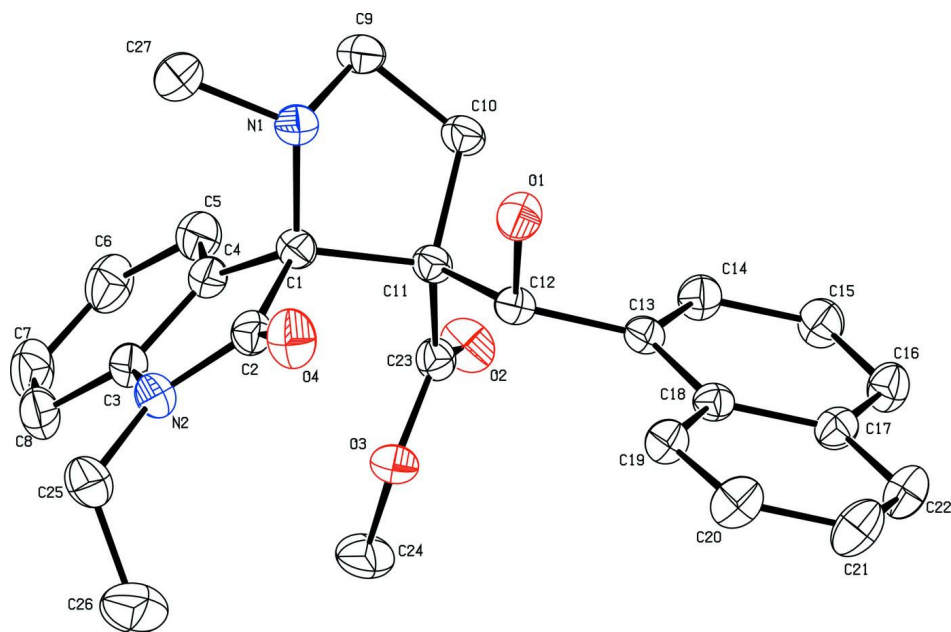
Spiro compounds have received considerable interest due to their biological properties (Pradhan *et al.*, 2006). In addition, pyrrolidine derivatives are found to have anticonvulsant, antimicrobial and antifungal activities against various pathogens (Amal Raj *et al.*, 2003). In view of their importance, 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).

### S2. Experimental

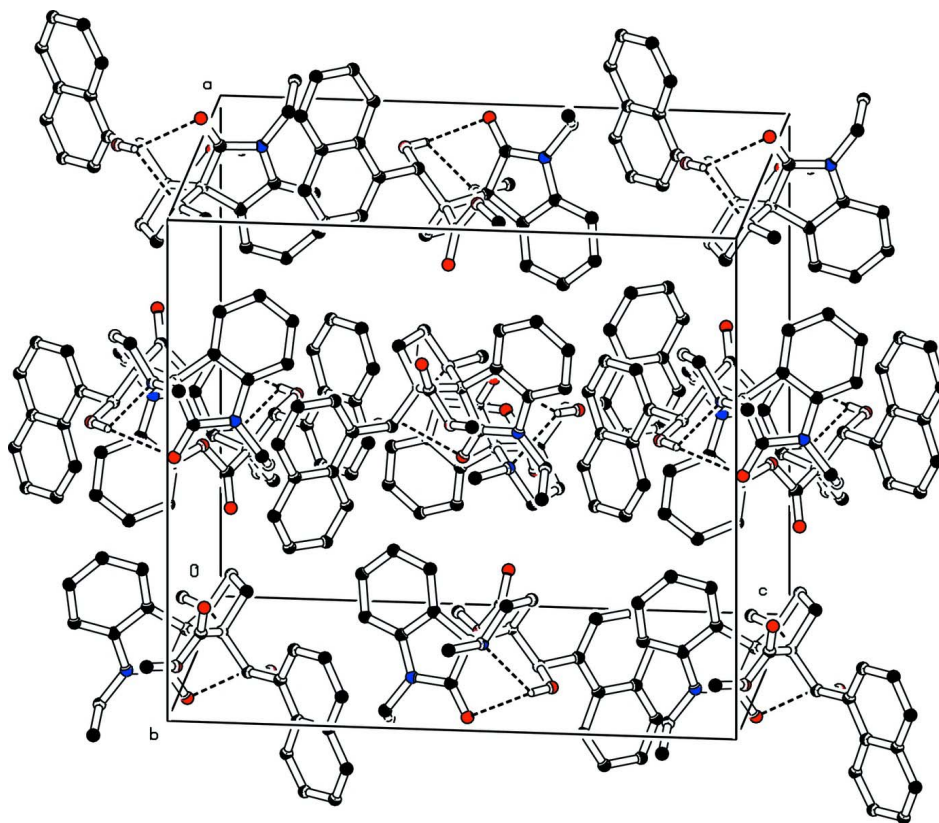
A mixture of methyl 2-(hydroxy(naphthalen-1-yl)methyl)acrylate (1 mmol), *N*-ethyl isatin (1.1 mmol) and sarcosine (1.1 mmol) was refluxed in methanol until completion of the reaction was evidenced by TLC analysis. After completion of the reaction the solvent was evaporated under reduced pressure. The 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

### S3. Refinement

All H atoms were positioned geometrically, with C—H = 0.93–0.97 Å and constrained to ride on their parent atom with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O},\text{C})$  for methyl H atoms and  $1.2U_{\text{eq}}(\text{C})$  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* $C_{27}H_{28}N_2O_4$  $M_r = 444.51$ Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

 $a = 16.7802 (3) \text{ \AA}$  $b = 14.6690 (3) \text{ \AA}$  $c = 18.4735 (4) \text{ \AA}$  $V = 4547.23 (16) \text{ \AA}^3$  $Z = 8$  $F(000) = 1888$  $D_x = 1.299 \text{ Mg m}^{-3}$ Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$ 

Cell parameters from 8834 reflections

 $\theta = 2.1\text{--}31.2^\circ$  $\mu = 0.09 \text{ mm}^{-1}$  $T = 293 \text{ K}$ 

Block, colourless

 $0.25 \times 0.20 \times 0.20 \text{ mm}$ *Data collection*

Bruker Kappa APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega$  and  $\phi$  scan

Absorption correction: multi-scan

(SADABS; Bruker, 2004)

 $T_{\min} = 0.979$ ,  $T_{\max} = 0.983$ 

44640 measured reflections

4636 independent reflections

3429 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.041$  $\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.2^\circ$  $h = -20 \rightarrow 18$  $k = -18 \rightarrow 18$  $l = -23 \rightarrow 23$

*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 methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 1.1973P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL97* (Sheldrick,  
2008),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$ 

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 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 ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>     | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|-------------|----------------------------------|
| 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       | 0.2892       | 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  | 0.86884 (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*     |
| C14  | 0.87276 (9)  | 0.41091 (11) | 0.28530 (8)  | 0.0415 (4) |
| H14  | 0.8245       | 0.3943       | 0.3063       | 0.050*     |
| C20  | 1.15904 (9)  | 0.39227 (12) | 0.26867 (10) | 0.0503 (4) |
| H20  | 1.2075       | 0.3688       | 0.2844       | 0.060*     |
| C2   | 0.96610 (9)  | 0.23489 (11) | 0.52364 (8)  | 0.0417 (4) |
| C9   | 0.79001 (10) | 0.16118 (11) | 0.41925 (9)  | 0.0471 (4) |
| H9A  | 0.7437       | 0.1598       | 0.4507       | 0.057*     |
| H9B  | 0.7845       | 0.1139       | 0.3829       | 0.057*     |
| C8   | 0.85759 (13) | 0.31094 (12) | 0.68131 (9)  | 0.0583 (5) |
| H8   | 0.8926       | 0.3189       | 0.7198       | 0.070*     |
| C21  | 1.15740 (10) | 0.45237 (12) | 0.20990 (10) | 0.0569 (5) |
| H21  | 1.2046       | 0.4686       | 0.1869       | 0.068*     |
| C5   | 0.75264 (10) | 0.28726 (12) | 0.56506 (9)  | 0.0490 (4) |
| H5   | 0.7175       | 0.2798       | 0.5266       | 0.059*     |
| C22  | 1.08714 (10) | 0.48694 (12) | 0.18645 (9)  | 0.0500 (4) |
| H22  | 1.0866       | 0.5272       | 0.1475       | 0.060*     |
| C27  | 0.85972 (13) | 0.07413 (12) | 0.51293 (10) | 0.0628 (5) |
| H27A | 0.9099       | 0.0696       | 0.5376       | 0.094*     |
| H27B | 0.8489       | 0.0180       | 0.4880       | 0.094*     |
| H27C | 0.8182       | 0.0857       | 0.5474       | 0.094*     |
| C24  | 0.89186 (12) | 0.50715 (12) | 0.53604 (10) | 0.0605 (5) |
| H24A | 0.9391       | 0.5241       | 0.5620       | 0.091*     |
| H24B | 0.8502       | 0.4928       | 0.5698       | 0.091*     |
| H24C | 0.8753       | 0.5569       | 0.5058       | 0.091*     |
| C6   | 0.72542 (12) | 0.31771 (13) | 0.63193 (10) | 0.0595 (5) |
| H6   | 0.6716       | 0.3305       | 0.6383       | 0.071*     |
| C25  | 1.03135 (11) | 0.26205 (13) | 0.64167 (10) | 0.0607 (5) |
| H25A | 1.0698       | 0.2172       | 0.6252       | 0.073*     |
| H25B | 1.0150       | 0.2452       | 0.6902       | 0.073*     |
| C15  | 0.87216 (10) | 0.47049 (11) | 0.22601 (9)  | 0.0471 (4) |
| H15  | 0.8239       | 0.4924       | 0.2084       | 0.057*     |
| C7   | 0.77716 (14) | 0.32907 (13) | 0.68843 (10) | 0.0647 (5) |
| H7   | 0.7577       | 0.3495       | 0.7327       | 0.078*     |
| C26  | 1.07060 (14) | 0.35314 (17) | 0.64422 (14) | 0.0871 (7) |
| H26A | 1.1159       | 0.3505       | 0.6759       | 0.131*     |
| H26B | 1.0334       | 0.3976       | 0.6619       | 0.131*     |
| H26C | 1.0877       | 0.3699       | 0.5965       | 0.131*     |

Atomic displacement parameters ( $\text{\AA}^2$ )

|    | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|----|------------|------------|------------|-------------|-------------|-------------|
| 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)  |
| C17 | 0.0396 (8)  | 0.0348 (8)  | 0.0421 (8)  | 0.0004 (6)   | 0.0047 (7)   | -0.0007 (7)  |
| C3  | 0.0564 (10) | 0.0392 (9)  | 0.0339 (8)  | -0.0044 (7)  | -0.0014 (7)  | 0.0047 (6)   |
| C1  | 0.0372 (8)  | 0.0376 (8)  | 0.0331 (7)  | -0.0024 (6)  | -0.0001 (6)  | -0.0020 (6)  |
| C4  | 0.0482 (9)  | 0.0374 (8)  | 0.0336 (7)  | -0.0064 (7)  | 0.0031 (7)   | 0.0016 (6)   |
| C10 | 0.0358 (8)  | 0.0504 (9)  | 0.0367 (8)  | -0.0091 (7)  | -0.0033 (6)  | -0.0035 (7)  |
| C19 | 0.0349 (8)  | 0.0406 (8)  | 0.0431 (8)  | 0.0018 (6)   | 0.0023 (6)   | 0.0022 (7)   |
| C11 | 0.0305 (7)  | 0.0377 (8)  | 0.0316 (7)  | -0.0027 (6)  | -0.0013 (6)  | -0.0024 (6)  |
| C23 | 0.0349 (8)  | 0.0412 (8)  | 0.0330 (7)  | 0.0003 (6)   | 0.0017 (6)   | 0.0004 (6)   |
| C16 | 0.0464 (10) | 0.0457 (9)  | 0.0454 (9)  | 0.0022 (7)   | -0.0011 (7)  | 0.0107 (7)   |
| C14 | 0.0317 (8)  | 0.0492 (9)  | 0.0434 (8)  | -0.0022 (6)  | 0.0006 (6)   | 0.0042 (7)   |
| C20 | 0.0327 (8)  | 0.0506 (10) | 0.0677 (11) | 0.0055 (7)   | 0.0058 (8)   | 0.0065 (8)   |
| C2  | 0.0435 (9)  | 0.0415 (9)  | 0.0402 (8)  | -0.0013 (7)  | -0.0030 (7)  | 0.0068 (7)   |
| C9  | 0.0492 (10) | 0.0460 (9)  | 0.0461 (9)  | -0.0123 (7)  | -0.0028 (7)  | -0.0066 (7)  |
| C8  | 0.0876 (15) | 0.0544 (11) | 0.0328 (8)  | -0.0036 (10) | -0.0018 (9)  | 0.0025 (8)   |
| C21 | 0.0402 (10) | 0.0541 (11) | 0.0765 (12) | 0.0031 (8)   | 0.0196 (9)   | 0.0147 (9)   |
| C5  | 0.0477 (10) | 0.0547 (10) | 0.0447 (9)  | -0.0037 (7)  | 0.0072 (7)   | 0.0012 (8)   |
| C22 | 0.0493 (10) | 0.0453 (10) | 0.0553 (10) | 0.0024 (7)   | 0.0137 (8)   | 0.0113 (8)   |
| C27 | 0.0863 (15) | 0.0439 (10) | 0.0583 (11) | -0.0106 (9)  | -0.0059 (10) | 0.0068 (8)   |
| C24 | 0.0749 (13) | 0.0447 (10) | 0.0621 (11) | -0.0021 (9)  | -0.0012 (10) | -0.0205 (9)  |
| C6  | 0.0653 (12) | 0.0578 (11) | 0.0553 (11) | 0.0042 (9)   | 0.0214 (9)   | 0.0052 (9)   |
| C25 | 0.0649 (12) | 0.0647 (12) | 0.0525 (10) | -0.0033 (9)  | -0.0249 (9)  | 0.0088 (9)   |
| C15 | 0.0379 (9)  | 0.0528 (10) | 0.0507 (9)  | 0.0029 (7)   | -0.0083 (7)  | 0.0086 (8)   |
| C7  | 0.0926 (16) | 0.0620 (12) | 0.0394 (9)  | 0.0073 (11)  | 0.0194 (10)  | 0.0017 (8)   |
| C26 | 0.0750 (15) | 0.0904 (17) | 0.0959 (17) | -0.0253 (12) | -0.0234 (13) | -0.0054 (14) |

*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—C23  | 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)   |
| C1—C4       | 1.523 (2)   | C6—H6         | 0.9300      |
| C1—C2       | 1.542 (2)   | C25—C26       | 1.490 (3)   |
| C1—C11      | 1.581 (2)   | C25—H25A      | 0.9700      |
| C4—C5       | 1.378 (2)   | C25—H25B      | 0.9700      |
| C10—C9      | 1.519 (2)   | C15—H15       | 0.9300      |
| C10—C11     | 1.5501 (19) | C7—H7         | 0.9300      |
| C10—H10A    | 0.9700      | C26—H26A      | 0.9600      |
| C10—H10B    | 0.9700      | C26—H26B      | 0.9600      |
| C19—C20     | 1.358 (2)   | C26—H26C      | 0.9600      |
| 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   | 108.6        | 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        | N2—C25—H25B     | 108.9        |
| C23—C11—C10     | 113.72 (12)  | C26—C25—H25B    | 108.9        |
| C23—C11—C12     | 108.73 (11)  | H25A—C25—H25B   | 107.8        |
| C10—C11—C12     | 112.50 (11)  | C16—C15—C14     | 120.62 (15)  |
| C23—C11—C1      | 107.70 (11)  | C16—C15—H15     | 119.7        |
| C10—C11—C1      | 101.67 (11)  | C14—C15—H15     | 119.7        |
| C12—C11—C1      | 112.35 (11)  | C6—C7—C8        | 121.59 (17)  |
| O2—C23—O3       | 123.65 (14)  | C6—C7—H7        | 119.2        |
| O2—C23—C11      | 126.86 (14)  | C8—C7—H7        | 119.2        |
| O3—C23—C11      | 109.47 (12)  | C25—C26—H26A    | 109.5        |
| C15—C16—C17     | 120.09 (14)  | C25—C26—H26B    | 109.5        |
| C15—C16—H16     | 120.0        | H26A—C26—H26B   | 109.5        |
| C17—C16—H16     | 120.0        | C25—C26—H26C    | 109.5        |
| C13—C14—C15     | 122.18 (14)  | H26A—C26—H26C   | 109.5        |
| C13—C14—H14     | 118.9        | H26B—C26—H26C   | 109.5        |
|                 |              |                 |              |
| 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—C12   | -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   | 31.45 (17)   |
| C18—C13—C12—O1  | -71.55 (15)  | C24—O3—C23—O2   | 12.4 (2)     |
| C14—C13—C12—C11 | -18.3 (2)    | C24—O3—C23—C11  | -165.93 (13) |
| C18—C13—C12—C11 | 164.14 (12)  | C10—C11—C23—O2  | -5.6 (2)     |
| C19—C18—C17—C22 | -0.3 (2)     | C12—C11—C23—O2  | 120.61 (16)  |
| C13—C18—C17—C22 | -178.76 (14) | C1—C11—C23—O2   | -117.41 (16) |
| C19—C18—C17—C16 | 178.06 (14)  | C10—C11—C23—O3  | 172.73 (11)  |
| C13—C18—C17—C16 | -0.4 (2)     | C12—C11—C23—O3  | -61.09 (14)  |
| C2—N2—C3—C8     | -177.79 (16) | C1—C11—C23—O3   | 60.89 (14)   |
| C25—N2—C3—C8    | 3.1 (3)      | C22—C17—C16—C15 | 178.16 (16)  |
| C2—N2—C3—C4     | 0.98 (18)    | C18—C17—C16—C15 | -0.1 (2)     |
| C25—N2—C3—C4    | -178.11 (14) | C18—C13—C14—C15 | -0.3 (2)     |
| C27—N1—C1—C4    | -49.96 (19)  | C12—C13—C14—C15 | -177.87 (14) |
| C9—N1—C1—C4     | 77.10 (15)   | C18—C19—C20—C21 | 0.3 (3)      |
| C27—N1—C1—C2    | 63.76 (17)   | C3—N2—C2—O4     | -177.41 (15) |
| C9—N1—C1—C2     | -169.18 (12) | C25—N2—C2—O4    | 1.7 (3)      |
| C27—N1—C1—C11   | -172.81 (14) | C3—N2—C2—C1     | -0.36 (17)   |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C9—N1—C1—C11    | -45.76 (14)  | C25—N2—C2—C1    | 178.75 (14)  |
| C8—C3—C4—C5     | 0.2 (2)      | N1—C1—C2—O4     | 53.2 (2)     |
| N2—C3—C4—C5     | -178.66 (14) | C4—C1—C2—O4     | 176.71 (16)  |
| C8—C3—C4—C1     | 177.69 (15)  | C11—C1—C2—O4    | -60.5 (2)    |
| N2—C3—C4—C1     | -1.16 (17)   | N1—C1—C2—N2     | -123.80 (13) |
| N1—C1—C4—C5     | -64.6 (2)    | C4—C1—C2—N2     | -0.32 (15)   |
| C2—C1—C4—C5     | 177.95 (17)  | C11—C1—C2—N2    | 122.47 (13)  |
| 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) | C16—C17—C22—C21 | -177.74 (17) |
| C13—C12—C11—C23 | -55.04 (15)  | C18—C17—C22—C21 | 0.6 (3)      |
| O1—C12—C11—C10  | -50.22 (15)  | C4—C5—C6—C7     | 0.2 (3)      |
| C13—C12—C11—C10 | 71.84 (16)   | C2—N2—C25—C26   | 92.5 (2)     |
| O1—C12—C11—C1   | 63.80 (14)   | C3—N2—C25—C26   | -88.5 (2)    |
| C13—C12—C11—C1  | -174.14 (11) | C17—C16—C15—C14 | 0.5 (3)      |
| N1—C1—C11—C23   | 154.27 (11)  | C13—C14—C15—C16 | -0.2 (3)     |
| C4—C1—C11—C23   | 28.58 (15)   | C5—C6—C7—C8     | 0.0 (3)      |
| C2—C1—C11—C23   | -88.26 (14)  | C3—C8—C7—C6     | -0.1 (3)     |
| N1—C1—C11—C10   | 34.47 (13)   |                 |              |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-------------------------|-------------|---------------|-----------------------|-------------------------|
| O1—H1...O4              | 0.82        | 2.37          | 2.9121 (16)           | 124                     |
| O1—H1...N1              | 0.82        | 2.39          | 2.9439 (17)           | 126                     |