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Structure Reports

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(R)-(+)-Dimethyl[4-oxido-2-oxo-1-(1-phenylethyl)-1,2,5,6-tetrahydropyridin-3-yl]sulfonium

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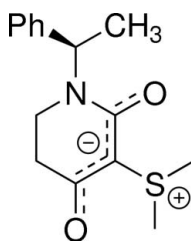
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.061; wR factor = 0.153; data-to-parameter ratio = 15.6.

In the title zwitterionic compound, $\text{C}_{15}\text{H}_{19}\text{NO}_2\text{S}$, the six-membered heterocycle adopts a sofa conformation. The negative charge is delocalized along the carbonyl and enolate system on the ring and the positive charge is localized on the S atom. Two intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions help to establish the packing.

Related literature

For background to the synthesis of chiral non-racemic zwitterionic 4-alkoxy-3-sulfonium ylide pyridine-2-ones, see: Zang *et al.* (2008); Kappe *et al.* (1983); Palillero *et al.* (2009). For the biological activity of related structures, see: Basco *et al.* (1994); Koruzñjak *et al.*, (2003). For ring conformation analysis, see: Cremer & Pople (1975).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{19}\text{NO}_2\text{S}$ $M_r = 277.37$ Orthorhombic, $P2_12_12_1$ $a = 5.9860$ (17) Å $b = 7.4050$ (14) Å $c = 31.589$ (5) Å $V = 1400.2$ (5) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.23$ mm⁻¹ $T = 293$ K $0.5 \times 0.4 \times 0.2$ mm

Data collection

Siemens P4 diffractometer
Absorption correction: ψ scan
(North *et al.*, 1968)
 $T_{\min} = 0.728$, $T_{\max} = 0.846$
3016 measured reflections
2683 independent reflections1928 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$
3 standard reflections every 97 reflections
intensity decay: 3%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.153$
 $S = 1.03$
2683 reflections
172 parameters
H-atom parameters constrained $\Delta\rho_{\text{max}} = 0.63$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³
Absolute structure: Flack (1983),
532 Friedel pairs
Flack parameter: -0.01 (16)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C7}-\text{H7C}\cdots\text{O2}^{\text{i}}$ | 0.96 | 2.36 | 3.315 (6) | 172 |
| $\text{C15}-\text{H15A}\cdots\text{O1}^{\text{ii}}$ | 0.96 | 2.38 | 3.167 (5) | 138 |

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

Data collection: XSCANS (Siemens, 1994); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SIR2004 (Burla *et al.*, 2005); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

We are grateful to CONACyT (Project 83185) for financial support. PGG also thanks CONACyT for a scholarship (169011). Special thanks go to Dr Marcos Flores (USAI-FQ-UNAM) for useful comments.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5438).

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supplementary materials

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(*R*)-(+)-Dimethyl[4-oxido-2-oxo-1-(1-phenylethyl)-1,2,5,6-tetrahydropyridin-3-yl]sulfonium

P. G. Gordillo, J. L. Terán, J. R. Juárez and A. Mendoza

Comment

The synthesis of chiral non racemic zwitterionic 4-alkoxy-3-sulfonium ylide pyridine-2-ones is an original area of interest in organic chemistry (Zang *et al.*, 2008; Kappe *et al.*, 1983) because they are useful for the synthesis of piperidine-2,4-dione and pyridine-2-one (Palillero *et al.*, 2009) compounds and because of their interesting biological properties (Basco *et al.*, 1994; Koruzňjak *et al.*, 2003).

The title compound **I**, features a zwitterionic molecule. The chiral centre shows an *R* configuration with $[\alpha]_D = +70.5$. The six member ring N1/C1/C2/C3/C4/C5 shows an sofa conformation with puckering parameters (Cremer & Pople, 1975) $Q = 0.465$ (4) Å, $\theta_2 = 119.7$ (5)°, $\varphi_2 = 103.2$ (6)°, $q_2 = 0.404$ (4) Å and $q_3 = -0.231$ (4) Å. The bond distances of N1—C1, N1—C5, C5—C4 and C4—C3 show typical values, so that C2—C3 distance shows a single double bond (1.415 (5) Å), while C1—C2 (1.455 (5) Å) distance is shorter than common sp^3 — sp^3 bonds, furthermore C3—O2 (1.244 (5) Å) and C1—O1 (1.250 (4) Å) distances are longer than related enolates and amide groups respectively these values suggest that negative charge is delocalized over O1/C1/C2/C3/O2 system and in the sulfonium group is located the positive charge in the zwitterion. Crystal packing is stabilized by two weak intermolecular C—H \cdots O interactions.

Experimental

The title compound, was obtained by an intramolecular cyclization reaction of (1'*R*)-(+)-{[(2-methoxycarbonyl-ethyl)-(1'-phenyl-ethyl)-carbamoyl]-methyl}-dimethyl-sulfonium; bromide (1 mmol), which was dissolved in CH₃CN (10 mL), treated with KOH (1.2 mmol) and stirred for two hours at room temperature. The resulting mixture was concentrated in vacuum and dissolved in ethyl acetate, filtered and concentrated giving the desired compound in 95%. Crystals were obtained from an ethyl acetate/diethylether solution; m.p. 139–140°C, $[\alpha]_D = +70.5$ (*c* 1.1, MeOH). IR (KBr) 1656 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ (p.p.m., *J* Hz): 1.51 (d, *J* = 7.2, 3H, Me), 2.32 (m, 2H), 2.90 (m, 1H), 2.98 (s, 3H, SMe), 3.00 (s, 3H, SMe), 3.16 (m, 1H), 5.94 (q, *J* = 7.2, 1H), 7.27–7.40 (m, 5H). ¹³C NMR (100 MHz, CDCl₃) δ p.p.m. 15.4, 26.0, 26.3, 36.3, 37.6, 48.9, 74.3, 126.5–141.0, 166.2, 187.5. HRMS (FAB): Calcd for C₁₅H₁₉NO₂S: 277.1124. Found: 277.1103.

Refinement

H atoms linked to C atoms were placed in geometrical idealized positions and refined as riding on their parent atoms, with C—H = 0.93–0.98 Å and with $U_{iso}(H) = 1.2 U_{eq}(C)$ or $U_{eq}(H) = 1.5 U_{eq}(C)$ for methyl groups.

Figures

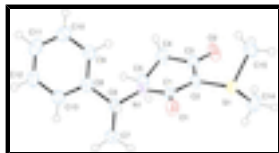


Fig. 1. The molecular structure of the title compound with 50% probability displacement ellipsoids for non-H atoms.

(R)-(+)-Dimethyl[4-oxido-2-oxo-1-(1-phenylethyl)-1,2,5,6-tetrahydropyridin-3-yl]sulfonium

Crystal data

$C_{15}H_{19}NO_2S$

$M_r = 277.37$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.9860$ (17) Å

$b = 7.4050$ (14) Å

$c = 31.589$ (5) Å

$V = 1400.2$ (5) Å³

$Z = 4$

$F(000) = 592$

$D_x = 1.316$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 46 reflections

$\theta = 21.3$ – 35.1°

$\mu = 0.23$ mm⁻¹

$T = 293$ K

Prism, colorless

$0.5 \times 0.4 \times 0.2$ mm

Data collection

Siemens P4
diffractometer

graphite

$2\theta/\omega$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.728$, $T_{\max} = 0.846$

3016 measured reflections

2683 independent reflections

1928 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 29.0^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -1 \rightarrow 8$

$k = -1 \rightarrow 10$

$l = -43 \rightarrow 1$

3 standard reflections every 97 reflections

intensity decay: 3%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.061$

$wR(F^2) = 0.153$

$S = 1.03$

2683 reflections

172 parameters

0 restraints

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 1.1965P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.63$ e Å⁻³

$\Delta\rho_{\min} = -0.39$ e Å⁻³

Absolute structure: Flack (1983), 532 Friedel pairs

Primary atom site location: structure-invariant direct methods Flack parameter: $-0.01 (16)$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| S1 | 1.10027 (19) | 0.87732 (14) | 0.03372 (3) | 0.0323 (2) |
| O1 | 0.8859 (6) | 0.5383 (4) | 0.05505 (8) | 0.0393 (7) |
| N1 | 0.7630 (6) | 0.6122 (4) | 0.12114 (10) | 0.0300 (7) |
| O2 | 1.0851 (7) | 1.1013 (4) | 0.11356 (10) | 0.0511 (9) |
| C9 | 0.8558 (7) | 0.3759 (6) | 0.19114 (12) | 0.0351 (9) |
| H9 | 0.9723 | 0.4434 | 0.1798 | 0.042* |
| C1 | 0.8724 (7) | 0.6511 (5) | 0.08437 (10) | 0.0278 (8) |
| C6 | 0.6341 (8) | 0.4423 (5) | 0.12418 (12) | 0.0345 (10) |
| H6 | 0.7011 | 0.3572 | 0.1041 | 0.041* |
| C4 | 0.8971 (9) | 0.8843 (6) | 0.15731 (11) | 0.0373 (9) |
| H4A | 1.0124 | 0.8199 | 0.1727 | 0.045* |
| H4B | 0.8501 | 0.9863 | 0.1744 | 0.045* |
| C3 | 0.9911 (8) | 0.9518 (6) | 0.11576 (12) | 0.0340 (9) |
| C8 | 0.6557 (7) | 0.3579 (5) | 0.16847 (12) | 0.0305 (9) |
| C10 | 0.8816 (9) | 0.2937 (6) | 0.23044 (13) | 0.0404 (10) |
| H10 | 1.0143 | 0.3077 | 0.2454 | 0.049* |
| C15 | 1.3821 (8) | 0.9447 (7) | 0.04512 (13) | 0.0427 (11) |
| H15A | 1.4586 | 0.9711 | 0.0191 | 0.064* |
| H15B | 1.4578 | 0.8487 | 0.0596 | 0.064* |
| H15C | 1.3805 | 1.0505 | 0.0627 | 0.064* |
| C2 | 0.9708 (7) | 0.8304 (5) | 0.08140 (11) | 0.0285 (9) |
| C5 | 0.7006 (8) | 0.7604 (6) | 0.14990 (12) | 0.0345 (10) |
| H5A | 0.6507 | 0.7108 | 0.1767 | 0.041* |
| H5B | 0.578 | 0.8286 | 0.1377 | 0.041* |
| C13 | 0.4867 (8) | 0.2558 (6) | 0.18589 (14) | 0.0396 (10) |
| H13 | 0.3523 | 0.2426 | 0.1714 | 0.048* |
| C12 | 0.5156 (9) | 0.1720 (6) | 0.22507 (14) | 0.0445 (11) |
| H12 | 0.401 | 0.1021 | 0.2363 | 0.053* |
| C11 | 0.7107 (9) | 0.1916 (6) | 0.24725 (14) | 0.0443 (12) |
| H11 | 0.7278 | 0.1363 | 0.2735 | 0.053* |
| C14 | 0.9890 (9) | 1.0887 (7) | 0.01517 (15) | 0.0544 (13) |

supplementary materials

| | | | | |
|------|------------|------------|--------------|-------------|
| H14A | 1.0569 | 1.1193 | -0.0114 | 0.082* |
| H14B | 1.0205 | 1.1817 | 0.0355 | 0.082* |
| H14C | 0.8304 | 1.078 | 0.0115 | 0.082* |
| C7 | 0.3927 (9) | 0.4733 (7) | 0.10987 (16) | 0.0524 (13) |
| H7A | 0.3922 | 0.5252 | 0.082 | 0.079* |
| H7B | 0.3199 | 0.5539 | 0.1293 | 0.079* |
| H7C | 0.3147 | 0.36 | 0.1094 | 0.079* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| S1 | 0.0354 (5) | 0.0314 (4) | 0.0300 (4) | -0.0059 (5) | 0.0034 (5) | 0.0035 (4) |
| O1 | 0.056 (2) | 0.0317 (14) | 0.0304 (13) | -0.0091 (19) | 0.0042 (15) | -0.0028 (11) |
| N1 | 0.0319 (17) | 0.0246 (15) | 0.0334 (15) | -0.0086 (18) | 0.0047 (14) | 0.0014 (14) |
| O2 | 0.066 (2) | 0.0325 (15) | 0.0543 (17) | -0.023 (2) | 0.0150 (19) | -0.0100 (13) |
| C9 | 0.034 (2) | 0.0334 (19) | 0.0379 (19) | -0.002 (2) | 0.0054 (17) | 0.0015 (18) |
| C1 | 0.030 (2) | 0.0288 (18) | 0.0245 (15) | -0.003 (2) | -0.0033 (16) | 0.0029 (14) |
| C6 | 0.040 (3) | 0.0282 (18) | 0.0352 (19) | -0.010 (2) | 0.0001 (19) | 0.0041 (16) |
| C4 | 0.052 (2) | 0.0315 (18) | 0.0287 (17) | -0.007 (3) | 0.009 (2) | -0.0067 (16) |
| C3 | 0.037 (2) | 0.0305 (19) | 0.0348 (19) | -0.003 (2) | 0.0052 (19) | -0.0037 (17) |
| C8 | 0.036 (2) | 0.0214 (17) | 0.0341 (18) | 0.0030 (19) | 0.0052 (17) | -0.0042 (15) |
| C10 | 0.042 (2) | 0.041 (2) | 0.039 (2) | 0.006 (3) | -0.001 (2) | -0.0008 (18) |
| C15 | 0.031 (2) | 0.055 (3) | 0.042 (2) | -0.006 (3) | 0.008 (2) | 0.001 (2) |
| C2 | 0.033 (2) | 0.0234 (17) | 0.0292 (17) | -0.0035 (18) | 0.0036 (16) | 0.0020 (14) |
| C5 | 0.042 (2) | 0.030 (2) | 0.0321 (19) | -0.002 (2) | 0.0077 (19) | -0.0005 (16) |
| C13 | 0.034 (2) | 0.037 (2) | 0.048 (2) | -0.003 (2) | 0.001 (2) | 0.0091 (19) |
| C12 | 0.046 (3) | 0.040 (2) | 0.048 (3) | -0.006 (3) | 0.012 (2) | 0.013 (2) |
| C11 | 0.057 (3) | 0.041 (2) | 0.035 (2) | 0.013 (3) | 0.009 (2) | 0.0050 (19) |
| C14 | 0.051 (3) | 0.057 (3) | 0.055 (3) | 0.004 (3) | 0.004 (2) | 0.026 (2) |
| C7 | 0.042 (3) | 0.057 (3) | 0.058 (3) | -0.016 (3) | -0.015 (3) | 0.019 (2) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|--------|-----------|----------|-----------|
| S1—C2 | 1.729 (4) | C8—C13 | 1.378 (6) |
| S1—C15 | 1.796 (5) | C10—C11 | 1.379 (7) |
| S1—C14 | 1.799 (5) | C10—H10 | 0.93 |
| O1—C1 | 1.250 (4) | C15—H15A | 0.96 |
| N1—C1 | 1.364 (5) | C15—H15B | 0.96 |
| N1—C5 | 1.473 (5) | C15—H15C | 0.96 |
| N1—C6 | 1.479 (5) | C5—H5A | 0.97 |
| O2—C3 | 1.244 (5) | C5—H5B | 0.97 |
| C9—C10 | 1.391 (6) | C13—C12 | 1.395 (6) |
| C9—C8 | 1.402 (6) | C13—H13 | 0.93 |
| C9—H9 | 0.93 | C12—C11 | 1.369 (7) |
| C1—C2 | 1.455 (5) | C12—H12 | 0.93 |
| C6—C7 | 1.531 (7) | C11—H11 | 0.93 |
| C6—C8 | 1.538 (5) | C14—H14A | 0.96 |
| C6—H6 | 0.98 | C14—H14B | 0.96 |
| C4—C5 | 1.510 (6) | C14—H14C | 0.96 |

| | | | |
|-------------|------------|---------------|------------|
| C4—C3 | 1.513 (5) | C7—H7A | 0.96 |
| C4—H4A | 0.97 | C7—H7B | 0.96 |
| C4—H4B | 0.97 | C7—H7C | 0.96 |
| C3—C2 | 1.415 (5) | | |
| C2—S1—C15 | 107.6 (2) | H15A—C15—H15B | 109.5 |
| C2—S1—C14 | 107.0 (2) | S1—C15—H15C | 109.5 |
| C15—S1—C14 | 99.9 (2) | H15A—C15—H15C | 109.5 |
| C1—N1—C5 | 119.4 (3) | H15B—C15—H15C | 109.5 |
| C1—N1—C6 | 119.0 (3) | C3—C2—C1 | 124.4 (3) |
| C5—N1—C6 | 117.5 (3) | C3—C2—S1 | 120.1 (3) |
| C10—C9—C8 | 120.6 (4) | C1—C2—S1 | 114.9 (3) |
| C10—C9—H9 | 119.7 | N1—C5—C4 | 110.5 (3) |
| C8—C9—H9 | 119.7 | N1—C5—H5A | 109.5 |
| O1—C1—N1 | 121.4 (3) | C4—C5—H5A | 109.5 |
| O1—C1—C2 | 122.4 (3) | N1—C5—H5B | 109.5 |
| N1—C1—C2 | 116.2 (3) | C4—C5—H5B | 109.5 |
| N1—C6—C7 | 110.2 (4) | H5A—C5—H5B | 108.1 |
| N1—C6—C8 | 111.2 (3) | C8—C13—C12 | 120.5 (4) |
| C7—C6—C8 | 114.1 (4) | C8—C13—H13 | 119.8 |
| N1—C6—H6 | 107 | C12—C13—H13 | 119.8 |
| C7—C6—H6 | 107 | C11—C12—C13 | 120.9 (4) |
| C8—C6—H6 | 107 | C11—C12—H12 | 119.6 |
| C5—C4—C3 | 110.8 (3) | C13—C12—H12 | 119.6 |
| C5—C4—H4A | 109.5 | C12—C11—C10 | 119.6 (4) |
| C3—C4—H4A | 109.5 | C12—C11—H11 | 120.2 |
| C5—C4—H4B | 109.5 | C10—C11—H11 | 120.2 |
| C3—C4—H4B | 109.5 | S1—C14—H14A | 109.5 |
| H4A—C4—H4B | 108.1 | S1—C14—H14B | 109.5 |
| O2—C3—C2 | 124.2 (4) | H14A—C14—H14B | 109.5 |
| O2—C3—C4 | 120.7 (4) | S1—C14—H14C | 109.5 |
| C2—C3—C4 | 115.1 (3) | H14A—C14—H14C | 109.5 |
| C13—C8—C9 | 118.4 (4) | H14B—C14—H14C | 109.5 |
| C13—C8—C6 | 121.6 (4) | C6—C7—H7A | 109.5 |
| C9—C8—C6 | 119.9 (4) | C6—C7—H7B | 109.5 |
| C11—C10—C9 | 120.1 (5) | H7A—C7—H7B | 109.5 |
| C11—C10—H10 | 120 | C6—C7—H7C | 109.5 |
| C9—C10—H10 | 120 | H7A—C7—H7C | 109.5 |
| S1—C15—H15A | 109.5 | H7B—C7—H7C | 109.5 |
| S1—C15—H15B | 109.5 | | |
| C5—N1—C1—O1 | -164.3 (4) | O2—C3—C2—S1 | 5.4 (7) |
| C6—N1—C1—O1 | -7.7 (6) | C4—C3—C2—S1 | -172.1 (3) |
| C5—N1—C1—C2 | 15.5 (5) | O1—C1—C2—C3 | -169.5 (4) |
| C6—N1—C1—C2 | 172.0 (3) | N1—C1—C2—C3 | 10.7 (6) |
| C1—N1—C6—C7 | -90.2 (5) | O1—C1—C2—S1 | 1.8 (5) |
| C5—N1—C6—C7 | 66.8 (5) | N1—C1—C2—S1 | -178.0 (3) |
| C1—N1—C6—C8 | 142.2 (4) | C15—S1—C2—C3 | 46.9 (4) |
| C5—N1—C6—C8 | -60.8 (5) | C14—S1—C2—C3 | -59.6 (4) |
| C5—C4—C3—O2 | 151.0 (4) | C15—S1—C2—C1 | -124.8 (3) |

supplementary materials

| | | | |
|---------------|------------|-----------------|-----------|
| C5—C4—C3—C2 | -31.4 (6) | C14—S1—C2—C1 | 128.7 (3) |
| C10—C9—C8—C13 | -0.6 (6) | C1—N1—C5—C4 | -48.4 (5) |
| C10—C9—C8—C6 | -177.2 (4) | C6—N1—C5—C4 | 154.7 (3) |
| N1—C6—C8—C13 | 149.8 (4) | C3—C4—C5—N1 | 54.5 (5) |
| C7—C6—C8—C13 | 24.4 (6) | C9—C8—C13—C12 | -0.3 (6) |
| N1—C6—C8—C9 | -33.6 (5) | C6—C8—C13—C12 | 176.3 (4) |
| C7—C6—C8—C9 | -159.1 (4) | C8—C13—C12—C11 | 0.9 (7) |
| C8—C9—C10—C11 | 0.8 (6) | C13—C12—C11—C10 | -0.7 (7) |
| O2—C3—C2—C1 | 176.3 (4) | C9—C10—C11—C12 | -0.2 (7) |
| C4—C3—C2—C1 | -1.2 (6) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C7—H7C \cdots O2 ⁱ | 0.96 | 2.36 | 3.315 (6) | 172 |
| C15—H15A \cdots O1 ⁱⁱ | 0.96 | 2.38 | 3.167 (5) | 138 |

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

Fig. 1

