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# (*S*)-(+)-4-(Oxiran-2-ylmethoxy)-9*H*-carbazole

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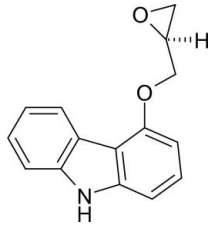
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.058; $wR$ factor = 0.124; data-to-parameter ratio = 8.0.

In the title compound, $C_{15}H_{13}NO_2$, all atoms of the carbazole group are coplanar (r.m.s. deviation = 0.005 Å), and the dihedral angle between this plane and C—O—C plane of oxane group is 57.1 (4)°. The crystal packing is stabilized by an N—H···O hydrogen bond, resulting in infinite supramolecular chains along [001].

## Related literature

For general background to the target product, see: Hildesheim *et al.* (2002); Morgan (1994). For other intermediates with similar structures, see: Herbert *et al.* (1987). For assignment of the absolute structure based on the synthesis, see: Rao *et al.* (2007)



## Experimental

### Crystal data

$C_{15}H_{13}NO_2$ $M_r$ = 239.26
Orthorhombic, $P2_12_12_1$
$a$ = 7.6140 (15) Å
$b$ = 9.5870 (19) Å
$c$ = 16.628 (3) Å
$V$ = 1213.8 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
0.30 × 0.10 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.974, $T_{max}$ = 0.991
2198 measured reflections
1298 independent reflections
834 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.061
3 standard reflections every 200 reflections
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.124
$S$ = 1.05
1298 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O2$^{i}$ | 0.86 | 2.09 | 2.948 (5) | 172 |

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge Professor Hua-qin Wang of the Analysis Center, Nanjing University, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2309).

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# supporting information

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## (*S*)-(+)-4-(Oxiran-2-ylmethoxy)-9*H*-carbazole

**Ding-Qiang Lu, Jia Chen, Wen-Yuan Wu, Xiu-Quan Ling and Ya-Jun Chang**

### S1. Comment

4-(2,3-epoxypropoxy)carbazole is used as a starting agent for the synthesis of 1-(carbazol-4-yloxy-3-[[2-(*O*-methoxyphenoxy)ethyl]amino]-2-propranol (Herbert & Heppenheim, 1987; Hildesheim *et al.*, 2002), which is a commercial drug (carvedilol) with $\alpha$- and $\beta_1$– receptor blocking activity that has been approved for the treatment of congestive heart failure (CHF). However carvedilol is actually a racemic mixture of the *R* and S enantiomers, and the $\beta$-receptor blocking activity of the S-enantiomer is about 200 times higher than that of *R*-carvedilol (Morgan, 1994).

We have now synthesized the title compound (CAS:67843–74-7), (I), as an intermediate in the synthesis of the target molecule, S-carvedilol, and report its structure here. The optically pure (*R*)-(-)-epichlorohydrin (CAS: 51594–55-9) was used as the starting agent, and during the reaction, an inversion of the chiral C atom occurred to give the final product (I) (Rao *et al.*, 2007).

Both the carbazole group and oxane group are planar, and the dihedral angle between them is 57.1 (4). The molecules are stacked along the *a* axis, and linked by N–H..O hydrogen bonds to form infinite chains along the [001] direction,

### S2. Experimental

For the preparation of the title compound, $K_2CO_3$ (20.73 g, 0.15 mol) and (*R*)-(-)-epichlorohydrin (7 ml, 0.09 mol) were added to an IPA (60 ml) solution containing 4-hydroxycarbazole (10.98 g, 0.06 mol). Then the reaction mixture was refluxed for 5 h at 355 K. The crude product was purified by recrystallization from ethyl acetate to provide colourless crystals suitable for X-ray analysis.

### S3. Refinement

H atoms were positioned geometrically [N–H = 0.86 Å, and C–H = 0.93, 0.97 and 0.98 Å for aromatic, methyne and methine H atoms, respectively] and constrained to ride on their parent atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(C or N). In the absence of significant anomalous scattering effects, Friedel pairs were merged for the final cycles of refinement.

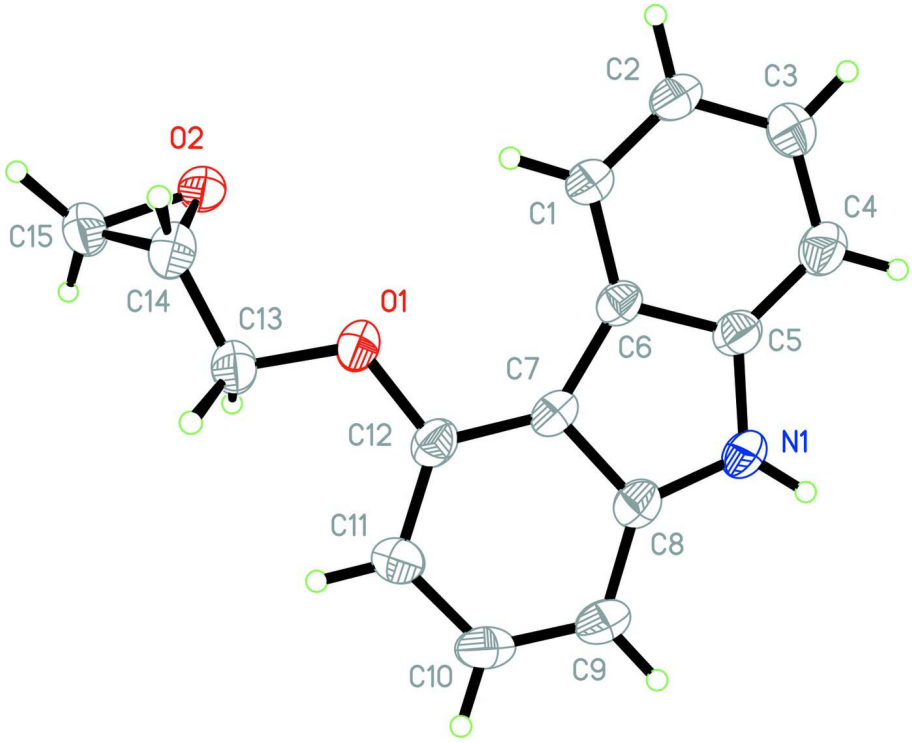


**Figure 1**

The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

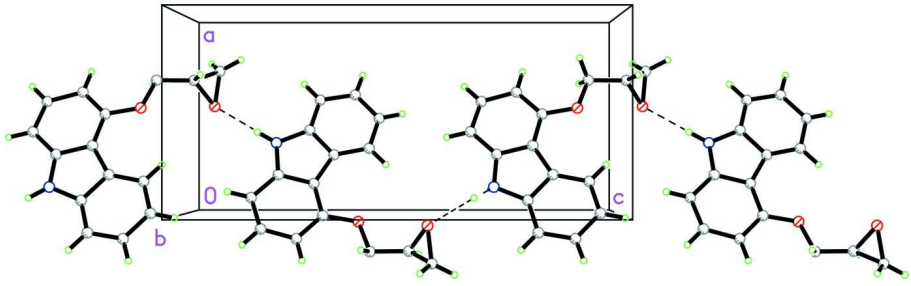


**Figure 2**

Supramolecular chains along the [010] direction by N–H···O hydrogen bonds (dashed lines).

### (*S*)-(+)-4-(Oxiran-2-ylmethoxy)-9*H*-carbazole

*Crystal data*

$C_{15}H_{13}NO_2$
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Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.6140\ (15)$ Å
$b = 9.5870\ (19)$ Å
$c = 16.628\ (3)$ Å
$V = 1213.8\ (4)$ Å$^3$
$Z = 4$
$F(000) = 504$
$D_x = 1.309$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.09$ mm$^{-1}$
$T = 293$ K
Prism, colourless
0.30 × 0.10 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.974, $T_{max}$ = 0.991
2198 measured reflections
1298 independent reflections
834 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.061
$\theta_{max}$ = 25.4°, $\theta_{min}$ = 2.5°
$h = -9\rightarrow9$
$k = 0\rightarrow11$
$l = 0\rightarrow19$
3 standard reflections every 200 reflections
intensity decay: none

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.124
$S$ = 1.05
1298 reflections
163 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
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.3329P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.8591 (5) | 1.1011 (4) | 0.7894 (2) | 0.0555 (11) |
| H1 | 0.9127 | 1.1158 | 0.8341 | 0.067* |
| O1 | 0.4664 (4) | 0.9701 (4) | 0.59614 (16) | 0.0626 (10) |
| O2 | 0.4551 (4) | 0.8193 (4) | 0.43812 (17) | 0.0717 (11) |
| C1 | 0.8311 (6) | 1.1218 (5) | 0.5757 (2) | 0.0521 (12) |
| H1A | 0.7493 | 1.0967 | 0.5367 | 0.063* |
| C2 | 0.9839 (7) | 1.1859 (6) | 0.5539 (3) | 0.0659 (15) |
| H2A | 1.0065 | 1.2022 | 0.4998 | 0.079* |
| C3 | 1.1059 (7) | 1.2271 (6) | 0.6109 (3) | 0.0740 (16) |
| H3A | 1.2082 | 1.2719 | 0.5946 | 0.089* |
| C4 | 1.0774 (6) | 1.2024 (6) | 0.6922 (3) | 0.0652 (15) |
| H4A | 1.1595 | 1.2291 | 0.7306 | 0.078* |
| C5 | 0.9233 (6) | 1.1369 (5) | 0.7139 (3) | 0.0523 (12) |
| C6 | 0.7981 (6) | 1.0942 (5) | 0.6563 (2) | 0.0441 (11) |
| C7 | 0.6551 (6) | 1.0313 (5) | 0.6998 (2) | 0.0452 (11) |

| | | | | |
|---|---|---|---|---|
| C8 | 0.6986 (6) | 1.0394 (5) | 0.7821 (3) | 0.0520 (12) |
| C9 | 0.5853 (7) | 0.9866 (5) | 0.8409 (2) | 0.0595 (14) |
| H9A | 0.6131 | 0.9928 | 0.8953 | 0.071* |
| C10 | 0.4327 (7) | 0.9258 (6) | 0.8160 (3) | 0.0648 (15) |
| H10A | 0.3572 | 0.8893 | 0.8546 | 0.078* |
| C11 | 0.3850 (7) | 0.9158 (6) | 0.7348 (3) | 0.0651 (15) |
| H11A | 0.2803 | 0.8731 | 0.7198 | 0.078* |
| C12 | 0.4970 (6) | 0.9709 (5) | 0.6776 (2) | 0.0501 (12) |
| C13 | 0.3242 (6) | 0.8870 (6) | 0.5679 (2) | 0.0648 (15) |
| H13A | 0.2137 | 0.9217 | 0.5890 | 0.078* |
| H13B | 0.3387 | 0.7909 | 0.5849 | 0.078* |
| C14 | 0.3258 (7) | 0.8962 (7) | 0.4805 (3) | 0.0706 (15) |
| H14A | 0.3019 | 0.9894 | 0.4589 | 0.085* |
| C15 | 0.2734 (6) | 0.7819 (6) | 0.4288 (3) | 0.0687 (16) |
| H15A | 0.2331 | 0.6966 | 0.4541 | 0.082* |
| H15B | 0.2164 | 0.8046 | 0.3783 | 0.082* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.057 (2) | 0.073 (3) | 0.0360 (19) | −0.006 (2) | −0.0127 (19) | −0.0015 (19) |
| O1 | 0.0521 (18) | 0.092 (3) | 0.0436 (17) | −0.012 (2) | −0.0062 (15) | −0.0092 (17) |
| O2 | 0.053 (2) | 0.115 (3) | 0.0468 (19) | −0.007 (2) | 0.0064 (17) | −0.0009 (19) |
| C1 | 0.055 (3) | 0.059 (3) | 0.042 (2) | −0.002 (3) | −0.006 (2) | 0.004 (2) |
| C2 | 0.068 (4) | 0.090 (4) | 0.040 (3) | −0.011 (4) | −0.003 (3) | 0.005 (3) |
| C3 | 0.065 (3) | 0.093 (4) | 0.064 (3) | −0.025 (3) | 0.002 (3) | −0.003 (3) |
| C4 | 0.061 (3) | 0.080 (4) | 0.055 (3) | −0.008 (3) | −0.012 (3) | −0.004 (3) |
| C5 | 0.053 (3) | 0.061 (3) | 0.043 (2) | 0.000 (3) | −0.001 (2) | 0.000 (2) |
| C6 | 0.045 (3) | 0.050 (3) | 0.037 (2) | 0.004 (2) | −0.003 (2) | 0.001 (2) |
| C7 | 0.058 (3) | 0.042 (3) | 0.035 (2) | 0.007 (3) | −0.005 (2) | −0.004 (2) |
| C8 | 0.054 (3) | 0.057 (3) | 0.046 (2) | 0.004 (3) | −0.005 (2) | −0.003 (2) |
| C9 | 0.071 (4) | 0.070 (4) | 0.037 (2) | 0.005 (3) | 0.004 (2) | 0.002 (2) |
| C10 | 0.076 (4) | 0.070 (4) | 0.048 (3) | 0.000 (3) | 0.021 (3) | 0.002 (3) |
| C11 | 0.059 (3) | 0.079 (4) | 0.058 (3) | −0.014 (3) | 0.015 (3) | −0.005 (3) |
| C12 | 0.050 (3) | 0.058 (3) | 0.043 (2) | 0.007 (3) | −0.004 (2) | −0.011 (2) |
| C13 | 0.045 (3) | 0.092 (4) | 0.057 (3) | −0.007 (3) | −0.002 (2) | −0.012 (3) |
| C14 | 0.058 (3) | 0.093 (4) | 0.060 (3) | −0.007 (3) | −0.011 (3) | −0.003 (3) |
| C15 | 0.056 (3) | 0.084 (4) | 0.066 (3) | −0.015 (3) | −0.003 (3) | −0.008 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C8 | 1.363 (6) | C6—C7 | 1.439 (6) |
| N1—C5 | 1.389 (6) | C7—C12 | 1.386 (6) |
| N1—H1 | 0.8600 | C7—C8 | 1.409 (5) |
| O1—C12 | 1.375 (4) | C8—C9 | 1.399 (6) |
| O1—C13 | 1.424 (5) | C9—C10 | 1.364 (7) |
| O2—C14 | 1.418 (6) | C9—H9A | 0.9300 |
| O2—C15 | 1.438 (6) | C10—C11 | 1.401 (6) |

| | | | |
|---|---|---|---|
| C1—C2 | 1.365 (7) | C10—H10A | 0.9300 |
| C1—C6 | 1.389 (5) | C11—C12 | 1.382 (6) |
| C1—H1A | 0.9300 | C11—H11A | 0.9300 |
| C2—C3 | 1.385 (6) | C13—C14 | 1.455 (6) |
| C2—H2A | 0.9300 | C13—H13A | 0.9700 |
| C3—C4 | 1.388 (6) | C13—H13B | 0.9700 |
| C3—H3A | 0.9300 | C14—C15 | 1.449 (7) |
| C4—C5 | 1.380 (6) | C14—H14A | 0.9800 |
| C4—H4A | 0.9300 | C15—H15A | 0.9700 |
| C5—C6 | 1.412 (6) | C15—H15B | 0.9700 |

| | | | |
|---|---|---|---|
| C8—N1—C5 | 110.0 (4) | C10—C9—H9A | 121.1 |
| C8—N1—H1 | 125.0 | C8—C9—H9A | 121.1 |
| C5—N1—H1 | 125.0 | C9—C10—C11 | 122.9 (5) |
| C12—O1—C13 | 117.2 (4) | C9—C10—H10A | 118.6 |
| C14—O2—C15 | 61.0 (3) | C11—C10—H10A | 118.6 |
| C2—C1—C6 | 119.7 (4) | C12—C11—C10 | 118.4 (5) |
| C2—C1—H1A | 120.1 | C12—C11—H11A | 120.8 |
| C6—C1—H1A | 120.1 | C10—C11—H11A | 120.8 |
| C1—C2—C3 | 121.2 (4) | O1—C12—C11 | 124.9 (4) |
| C1—C2—H2A | 119.4 | O1—C12—C7 | 114.3 (4) |
| C3—C2—H2A | 119.4 | C11—C12—C7 | 120.8 (4) |
| C2—C3—C4 | 120.8 (5) | O1—C13—C14 | 106.8 (4) |
| C2—C3—H3A | 119.6 | O1—C13—H13A | 110.4 |
| C4—C3—H3A | 119.6 | C14—C13—H13A | 110.4 |
| C5—C4—C3 | 117.8 (4) | O1—C13—H13B | 110.4 |
| C5—C4—H4A | 121.1 | C14—C13—H13B | 110.4 |
| C3—C4—H4A | 121.1 | H13A—C13—H13B | 108.6 |
| C4—C5—N1 | 130.4 (4) | O2—C14—C15 | 60.2 (3) |
| C4—C5—C6 | 121.9 (4) | O2—C14—C13 | 118.1 (5) |
| N1—C5—C6 | 107.7 (4) | C15—C14—C13 | 123.0 (5) |
| C1—C6—C5 | 118.5 (4) | O2—C14—H14A | 114.8 |
| C1—C6—C7 | 134.5 (4) | C15—C14—H14A | 114.8 |
| C5—C6—C7 | 106.9 (3) | C13—C14—H14A | 114.8 |
| C12—C7—C8 | 119.0 (4) | O2—C15—C14 | 58.8 (3) |
| C12—C7—C6 | 134.3 (4) | O2—C15—H15A | 117.9 |
| C8—C7—C6 | 106.7 (4) | C14—C15—H15A | 117.9 |
| N1—C8—C9 | 130.3 (4) | O2—C15—H15B | 117.9 |
| N1—C8—C7 | 108.7 (4) | C14—C15—H15B | 117.9 |
| C9—C8—C7 | 121.0 (4) | H15A—C15—H15B | 115.0 |
| C10—C9—C8 | 117.8 (4) | | |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | −1.5 (8) | C6—C7—C8—N1 | −0.9 (5) |
| C1—C2—C3—C4 | 1.0 (9) | C12—C7—C8—C9 | 0.5 (7) |
| C2—C3—C4—C5 | −0.6 (9) | C6—C7—C8—C9 | −180.0 (4) |
| C3—C4—C5—N1 | −178.9 (5) | N1—C8—C9—C10 | −178.0 (5) |
| C3—C4—C5—C6 | 0.7 (8) | C7—C8—C9—C10 | 0.7 (7) |
| C8—N1—C5—C4 | 179.0 (5) | C8—C9—C10—C11 | −0.8 (8) |

| | | | |
|---|---|---|---|
| C8—N1—C5—C6 | −0.7 (5) | C9—C10—C11—C12 | −0.4 (8) |
| C2—C1—C6—C5 | 1.5 (7) | C13—O1—C12—C11 | −10.7 (7) |
| C2—C1—C6—C7 | 179.4 (5) | C13—O1—C12—C7 | 168.4 (4) |
| C4—C5—C6—C1 | −1.2 (7) | C10—C11—C12—O1 | −179.3 (5) |
| N1—C5—C6—C1 | 178.5 (4) | C10—C11—C12—C7 | 1.7 (8) |
| C4—C5—C6—C7 | −179.6 (4) | C8—C7—C12—O1 | 179.1 (4) |
| N1—C5—C6—C7 | 0.1 (5) | C6—C7—C12—O1 | −0.3 (8) |
| C1—C6—C7—C12 | 1.9 (10) | C8—C7—C12—C11 | −1.7 (8) |
| C5—C6—C7—C12 | 180.0 (5) | C6—C7—C12—C11 | 178.9 (5) |
| C1—C6—C7—C8 | −177.6 (5) | C12—O1—C13—C14 | −176.5 (4) |
| C5—C6—C7—C8 | 0.5 (5) | C15—O2—C14—C13 | 113.9 (6) |
| C5—N1—C8—C9 | 179.9 (5) | O1—C13—C14—O2 | 75.6 (6) |
| C5—N1—C8—C7 | 1.0 (5) | O1—C13—C14—C15 | 146.6 (5) |
| C12—C7—C8—N1 | 179.5 (4) | C13—C14—C15—O2 | −106.0 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O2$^{i}$ | 0.86 | 2.09 | 2.948 (5) | 172 |

Symmetry code: (i) −*x*+3/2, −*y*+2, *z*+1/2.