Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 1-[1-(Hydroxyimino)ethyl]-*N*-(2-methoxyphenyl)cyclopropanecarboxamide

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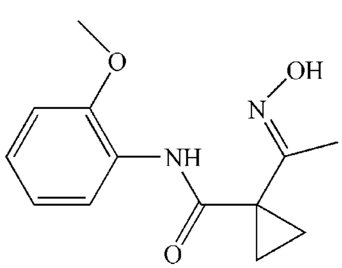
Received 30 May 2009; accepted 11 June 2009

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.073; $wR$ factor = 0.170; data-to-parameter ratio = 14.4.

The title compound, $C_{13}H_{16}N_2O_3$, adopts an $E$ configuration with respect to the C═N bond and an intramolecular N—H⋯N hydrogen bond results in the formation of a six-membered ring. In the crystal, intermolecular O—H⋯O hydrogen bonds link the molecules into a chain propagating along the $b$ axis. Very weak $\pi$–$\pi$ stacking interactions [centroid–centroid distance = 4.18 (2) Å] may further consolidate the packing, forming a two-dimensional supramolecular network.

## Related literature

For background to cyclopropane derivatives, see: Liu & Montgomery (2006); Ogoshi *et al.* (2006).




## Experimental

*Crystal data*

$C_{13}H_{16}N_2O_3$
$M_r$ = 248.28
Monoclinic, $P2_1/c$
$a$ = 16.062 (6) Å
$b$ = 5.483 (2) Å
$c$ = 14.250 (6) Å
$\beta$ = 100.055 (6)°
$V$ = 1235.7 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 293 K
0.41 × 0.29 × 0.20 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999) $T_{min}$ = 0.96, $T_{max}$ = 0.99
6430 measured reflections
2432 independent reflections
1520 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.044

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.170
$S$ = 1.09
2432 reflections
169 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N1—H1$N$⋯N2 | 0.856 (17) | 1.94 (2) | 2.670 (3) | 142 (3) |
| O3—H3$O$⋯O2$^{i}$ | 0.85 (4) | 1.93 (2) | 2.751 (3) | 162 (4) |

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1008); molecular graphics: *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

We thank the College of Chemical Engineering of Shanxi Datong University for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2995).

## References

Bruker (1999). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Liu, L. & Montgomery, J. (2006). *J. Am. Chem. Soc.* **128**, 5348–5349.
Ogoshi, S., Nagata, M. & Kurosawa, H. (2006). *J. Am. Chem. Soc.* **128**, 5350–5351.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2009). E**65**, o1618 [doi:10.1107/S1600536809022260]

## 1-[1-(Hydroxyimino)ethyl]-*N*-(2-methoxyphenyl)cyclopropanecarboxamide

**Jun-Ling Wang, Shuang-Ming Meng, Mao-Zhong Tian and Feng Feng**

### S1. Comment

Cyclopropane and their derivatives are a significant class of compounds which can be used in a variety of studies such as organic synthesis, catalytic reaction and so on (Liu & Montgomery, 2006; Ogoshi *et al.*, 2006). In order to extend our work on structural characterization of cyclopropane compounds, we report the synthesis and the X-ray structure of the title compound, (I), in this paper (Fig. 1).

The title molecule adopts an E configuration with respect to C=N bond. There is an intramolecular O—H···N hydrogen bonds, forming of a six-membered ring (Table 1) and the intermolecular O—H···O hydrogen bonds link the molecules into a one-dimensional chain along the *b* axis. The crystal structure is further stabilized by $\pi$-$\pi$ interaction involving the benzene rings: *Cg*1···*Cg*1 (1 - *x*, 1 - *y*, 1 - *z*) = 4.18 (2) Å, where *Cg*1 denotes the centroid of the C2—C7 (Fig. 2).

### S2. Experimental

To a solution of 1-acetyl-*N*-(2-methoxyphenyl)cyclopropanecarboxamide (2.33 g, 10 mmol) and NaOAc (1.64 g, 20 mmol) in EtOH (25 ml) and $H_2O$ (1 ml) was added $NH_2OH.HCl$ (1.39 g, 20 mmol) in one portion. The reaction mixture was stirred at room temperature for 12 h, and then poured into ice-water (200 ml) under stirring. A white solid was precipitated, which was filtered and the residue was purified by a flash silica gel column chromatography to give colourless blocks of (I) (eluent: ether/ethyl acetate = 1/3 v/v).

### S3. Refinement

The N- and O-bound H atoms were located in a difference map and their positions were freely refined. The C-bound H atoms were geometrically placed (C—H = 0.93–0.97Å) and refined as riding. The constraints $U_{iso}$ = 1.2$U$eq(C,N) or 1.5$U$eq(methyl C,O) were applied.

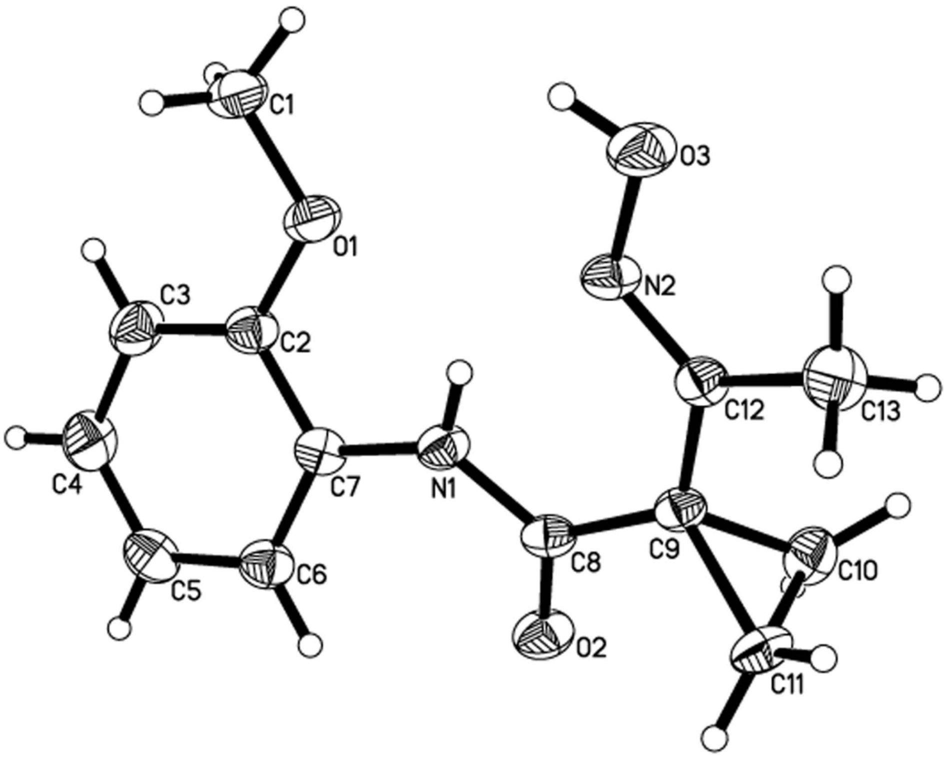



**Figure 1**

Molecule structure of (I) with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

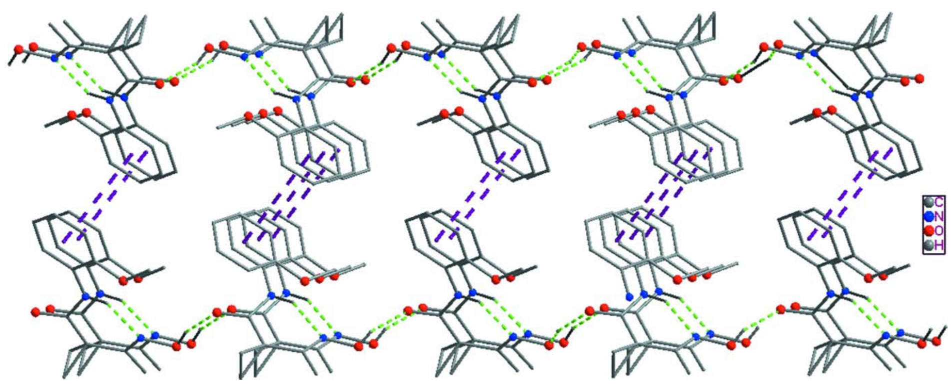



**Figure 2**

View of the two-dimensional supramolecular structure of (I): hydrogen bonds and $\pi$-$\pi$ interactions are shown as dashed lines.

### 1-[1-(Hydroxyimino)ethyl]-*N*-(2-methoxyphenyl)cyclopropanecarboxamide

*Crystal data*

$C_{13}H_{16}N_2O_3$
$M_r$ = 248.28
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 16.062 (6) Å
$b$ = 5.483 (2) Å
$c$ = 14.250 (6) Å
$\beta$ = 100.055 (6)°
$V$ = 1235.7 (8) Å$^3$
$Z$ = 4
$F$(000) = 528
$D_x$ = 1.335 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71069 Å
Cell parameters from 2432 reflections

$\theta = 1.3–26.1°$
$\mu = 0.10\ mm^{-1}$
$T = 293\ K$
Block, colourless
0.41 × 0.29 × 0.20 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.96$, $T_{max} = 0.99$
6430 measured reflections
2432 independent reflections
1520 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 26.1°$, $\theta_{min} = 1.3°$
$h = -9 \rightarrow 19$
$k = -6 \rightarrow 6$
$l = -17 \rightarrow 17$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.170$
$S = 1.09$
2432 reflections
169 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.566P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.3277 (2) | −0.2371 (7) | 1.1123 (2) | 0.0577 (10) |
| H1A | 0.2878 | −0.2249 | 1.1550 | 0.087* |
| H1B | 0.3826 | −0.1874 | 1.1447 | 0.087* |
| H1C | 0.3303 | −0.4029 | 1.0914 | 0.087* |
| C2 | 0.3509 (2) | −0.0767 (6) | 0.9634 (2) | 0.0428 (8) |
| C3 | 0.4169 (2) | −0.2323 (6) | 0.9573 (3) | 0.0558 (10) |
| H3 | 0.4305 | −0.3561 | 1.0020 | 0.067* |
| C4 | 0.4628 (2) | −0.2049 (7) | 0.8849 (3) | 0.0578 (10) |
| H4 | 0.5075 | −0.3099 | 0.8812 | 0.069* |
| C5 | 0.4432 (2) | −0.0246 (6) | 0.8182 (2) | 0.0525 (9) |
| H5 | 0.4745 | −0.0076 | 0.7696 | 0.063* |
| C6 | 0.3768 (2) | 0.1321 (6) | 0.8233 (2) | 0.0468 (8) |

| | | | | |
|---|---|---|---|---|
| H6 | 0.3637 | 0.2548 | 0.7781 | 0.056* |
| C7 | 0.3297 (2) | 0.1078 (5) | 0.8954 (2) | 0.0378 (7) |
| C8 | 0.21464 (19) | 0.4042 (5) | 0.8447 (2) | 0.0365 (7) |
| C9 | 0.14682 (19) | 0.5534 (5) | 0.8782 (2) | 0.0356 (7) |
| C10 | 0.0684 (2) | 0.5852 (6) | 0.8000 (2) | 0.0516 (9) |
| H10A | 0.0671 | 0.4975 | 0.7406 | 0.062* |
| H10B | 0.0139 | 0.6022 | 0.8198 | 0.062* |
| C11 | 0.1292 (2) | 0.7886 (6) | 0.8211 (2) | 0.0500 (9) |
| H11A | 0.1116 | 0.9299 | 0.8537 | 0.060* |
| H11B | 0.1647 | 0.8252 | 0.7745 | 0.060* |
| C12 | 0.13045 (19) | 0.5525 (5) | 0.9782 (2) | 0.0366 (7) |
| C13 | 0.0787 (2) | 0.7537 (6) | 1.0116 (3) | 0.0559 (10) |
| H13A | 0.0740 | 0.7263 | 1.0770 | 0.084* |
| H13B | 0.0233 | 0.7553 | 0.9731 | 0.084* |
| H13C | 0.1059 | 0.9076 | 1.0059 | 0.084* |
| N2 | 0.16185 (17) | 0.3782 (4) | 1.03227 (17) | 0.0405 (7) |
| O1 | 0.30181 (15) | −0.0837 (4) | 1.03230 (16) | 0.0589 (7) |
| O2 | 0.22453 (15) | 0.4164 (4) | 0.76124 (15) | 0.0523 (6) |
| O3 | 0.14111 (17) | 0.3914 (4) | 1.12368 (16) | 0.0580 (7) |
| H3O | 0.176 (2) | 0.300 (7) | 1.159 (3) | 0.087* |
| N1 | 0.26309 (17) | 0.2616 (4) | 0.90914 (18) | 0.0392 (7) |
| H1N | 0.2470 (19) | 0.254 (6) | 0.9632 (16) | 0.047* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.067 (3) | 0.060 (2) | 0.048 (2) | 0.008 (2) | 0.0146 (18) | 0.0174 (18) |
| C2 | 0.051 (2) | 0.0407 (17) | 0.0392 (18) | 0.0018 (16) | 0.0135 (15) | −0.0005 (15) |
| C3 | 0.061 (2) | 0.050 (2) | 0.057 (2) | 0.0181 (19) | 0.0139 (19) | 0.0073 (17) |
| C4 | 0.058 (2) | 0.056 (2) | 0.064 (3) | 0.0165 (19) | 0.0211 (19) | −0.0074 (19) |
| C5 | 0.055 (2) | 0.059 (2) | 0.049 (2) | 0.0000 (19) | 0.0237 (17) | −0.0093 (18) |
| C6 | 0.054 (2) | 0.0429 (18) | 0.046 (2) | −0.0002 (17) | 0.0155 (17) | −0.0008 (15) |
| C7 | 0.045 (2) | 0.0327 (16) | 0.0372 (17) | −0.0028 (15) | 0.0115 (14) | −0.0061 (14) |
| C8 | 0.046 (2) | 0.0311 (15) | 0.0317 (17) | −0.0094 (15) | 0.0058 (14) | −0.0012 (13) |
| C9 | 0.0413 (18) | 0.0258 (14) | 0.0394 (17) | −0.0050 (14) | 0.0064 (14) | 0.0022 (13) |
| C10 | 0.049 (2) | 0.053 (2) | 0.050 (2) | 0.0036 (18) | 0.0013 (16) | 0.0030 (17) |
| C11 | 0.065 (2) | 0.0338 (17) | 0.051 (2) | 0.0036 (17) | 0.0092 (18) | 0.0139 (15) |
| C12 | 0.0402 (19) | 0.0257 (14) | 0.0444 (18) | −0.0048 (14) | 0.0089 (14) | −0.0011 (13) |
| C13 | 0.068 (3) | 0.0383 (19) | 0.067 (3) | 0.0096 (18) | 0.026 (2) | −0.0046 (17) |
| N2 | 0.0562 (18) | 0.0345 (14) | 0.0336 (14) | 0.0018 (13) | 0.0154 (12) | 0.0017 (12) |
| O1 | 0.0641 (17) | 0.0655 (16) | 0.0524 (15) | 0.0226 (13) | 0.0245 (12) | 0.0235 (12) |
| O2 | 0.0684 (16) | 0.0570 (15) | 0.0329 (13) | 0.0035 (13) | 0.0128 (11) | 0.0033 (11) |
| O3 | 0.0794 (19) | 0.0602 (16) | 0.0402 (14) | 0.0181 (14) | 0.0264 (12) | 0.0069 (11) |
| N1 | 0.0509 (17) | 0.0354 (13) | 0.0334 (15) | 0.0070 (13) | 0.0133 (13) | 0.0018 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O1 | 1.420 (4) | C8—C9 | 1.505 (4) |
| C1—H1A | 0.9600 | C9—C12 | 1.494 (4) |
| C1—H1B | 0.9600 | C9—C11 | 1.525 (4) |
| C1—H1C | 0.9600 | C9—C10 | 1.540 (4) |
| C2—O1 | 1.362 (4) | C10—C11 | 1.478 (5) |
| C2—C3 | 1.376 (4) | C10—H10A | 0.9700 |
| C2—C7 | 1.402 (4) | C10—H10B | 0.9700 |
| C3—C4 | 1.377 (5) | C11—H11A | 0.9700 |
| C3—H3 | 0.9300 | C11—H11B | 0.9700 |
| C4—C5 | 1.369 (5) | C12—N2 | 1.275 (4) |
| C4—H4 | 0.9300 | C12—C13 | 1.507 (4) |
| C5—C6 | 1.380 (5) | C13—H13A | 0.9600 |
| C5—H5 | 0.9300 | C13—H13B | 0.9600 |
| C6—C7 | 1.385 (4) | C13—H13C | 0.9600 |
| C6—H6 | 0.9300 | N2—O3 | 1.402 (3) |
| C7—N1 | 1.402 (4) | O3—H3O | 0.85 (4) |
| C8—O2 | 1.229 (3) | N1—H1N | 0.856 (17) |
| C8—N1 | 1.345 (4) | | |
| | | | |
| O1—C1—H1A | 109.5 | C12—C9—C10 | 115.7 (3) |
| O1—C1—H1B | 109.5 | C8—C9—C10 | 112.2 (3) |
| H1A—C1—H1B | 109.5 | C11—C9—C10 | 57.7 (2) |
| O1—C1—H1C | 109.5 | C11—C10—C9 | 60.6 (2) |
| H1A—C1—H1C | 109.5 | C11—C10—H10A | 117.7 |
| H1B—C1—H1C | 109.5 | C9—C10—H10A | 117.7 |
| O1—C2—C3 | 125.3 (3) | C11—C10—H10B | 117.7 |
| O1—C2—C7 | 114.7 (3) | C9—C10—H10B | 117.7 |
| C3—C2—C7 | 120.0 (3) | H10A—C10—H10B | 114.8 |
| C2—C3—C4 | 120.0 (3) | C10—C11—C9 | 61.7 (2) |
| C2—C3—H3 | 120.0 | C10—C11—H11A | 117.6 |
| C4—C3—H3 | 120.0 | C9—C11—H11A | 117.6 |
| C5—C4—C3 | 120.7 (3) | C10—C11—H11B | 117.6 |
| C5—C4—H4 | 119.6 | C9—C11—H11B | 117.6 |
| C3—C4—H4 | 119.6 | H11A—C11—H11B | 114.7 |
| C4—C5—C6 | 119.9 (3) | N2—C12—C9 | 117.5 (3) |
| C4—C5—H5 | 120.0 | N2—C12—C13 | 122.7 (3) |
| C6—C5—H5 | 120.0 | C9—C12—C13 | 119.8 (3) |
| C5—C6—C7 | 120.4 (3) | C12—C13—H13A | 109.5 |
| C5—C6—H6 | 119.8 | C12—C13—H13B | 109.5 |
| C7—C6—H6 | 119.8 | H13A—C13—H13B | 109.5 |
| C6—C7—C2 | 119.0 (3) | C12—C13—H13C | 109.5 |
| C6—C7—N1 | 125.1 (3) | H13A—C13—H13C | 109.5 |
| C2—C7—N1 | 115.9 (3) | H13B—C13—H13C | 109.5 |
| O2—C8—N1 | 122.3 (3) | C12—N2—O3 | 112.9 (2) |
| O2—C8—C9 | 120.0 (3) | C2—O1—C1 | 118.0 (3) |
| N1—C8—C9 | 117.7 (2) | N2—O3—H3O | 107 (3) |

| | | | |
|---|---|---|---|
| C12—C9—C8 | 123.9 (3) | C8—N1—C7 | 128.2 (3) |
| C12—C9—C11 | 117.6 (3) | C8—N1—H1N | 114 (2) |
| C8—C9—C11 | 111.6 (3) | C7—N1—H1N | 117 (2) |
| | | | |
| O1—C2—C3—C4 | −179.1 (3) | C8—C9—C10—C11 | −102.3 (3) |
| C7—C2—C3—C4 | 0.8 (5) | C12—C9—C11—C10 | −104.3 (3) |
| C2—C3—C4—C5 | −0.4 (6) | C8—C9—C11—C10 | 103.3 (3) |
| C3—C4—C5—C6 | 0.1 (6) | C8—C9—C12—N2 | −16.4 (4) |
| C4—C5—C6—C7 | −0.2 (5) | C11—C9—C12—N2 | −165.1 (3) |
| C5—C6—C7—C2 | 0.6 (5) | C10—C9—C12—N2 | 129.6 (3) |
| C5—C6—C7—N1 | 177.7 (3) | C8—C9—C12—C13 | 163.4 (3) |
| O1—C2—C7—C6 | 179.0 (3) | C11—C9—C12—C13 | 14.7 (4) |
| C3—C2—C7—C6 | −0.9 (5) | C10—C9—C12—C13 | −50.6 (4) |
| O1—C2—C7—N1 | 1.7 (4) | C9—C12—N2—O3 | −178.6 (2) |
| C3—C2—C7—N1 | −178.2 (3) | C13—C12—N2—O3 | 1.7 (4) |
| O2—C8—C9—C12 | −179.5 (3) | C3—C2—O1—C1 | 10.3 (5) |
| N1—C8—C9—C12 | 0.1 (4) | C7—C2—O1—C1 | −169.6 (3) |
| O2—C8—C9—C11 | −29.2 (4) | O2—C8—N1—C7 | −0.4 (5) |
| N1—C8—C9—C11 | 150.4 (3) | C9—C8—N1—C7 | −179.9 (3) |
| O2—C8—C9—C10 | 33.5 (4) | C6—C7—N1—C8 | 23.1 (5) |
| N1—C8—C9—C10 | −146.9 (3) | C2—C7—N1—C8 | −159.8 (3) |
| C12—C9—C10—C11 | 107.8 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···N2 | 0.86 (2) | 1.94 (2) | 2.670 (3) | 142 (3) |
| O3—H3*O*···O2$^{i}$ | 0.85 (4) | 1.93 (2) | 2.751 (3) | 162 (4) |

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