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# 2-[(*E*)-3,4-Dimethoxybenzylidene]-hydrazinecarboxamide

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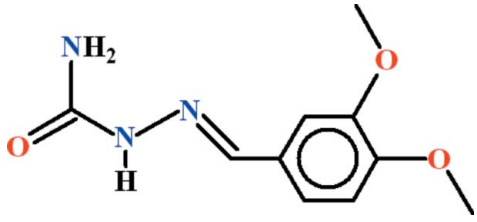
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.043; $wR$ factor = 0.121; data-to-parameter ratio = 14.4.

In the title compound, $C_{10}H_{13}N_3O_3$, the 3,4-dimethoxybenzylidene and hydrazinecarboxamide groups are oriented at a dihedral angle of 53.82 (6)° and an intramolecular N—H···N hydrogen bond generates an $S$(5) ring motif. In the crystal, molecules are linked by N—H···O hydrogen bonds into sheets propagating in ($\bar{2}$01), which feature $R_1^2(5)$, $R_2^2(8)$ and $R_2^4(14)$ loops.

## Related literature

For related structures, see: Fun *et al.* (2011); Liang *et al.* (2007); For graph-set notation, see: Bernstein *et al.* (1995).




## Experimental

### Crystal data

$C_{10}H_{13}N_3O_3$
$M_r$ = 223.23
Monoclinic, $C2/c$
$a$ = 22.2300 (7) Å
$b$ = 7.6367 (3) Å
$c$ = 15.6482 (6) Å
$\beta$ = 126.234 (1)°
$V$ = 2142.76 (14) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 296 K
0.25 × 0.18 × 0.15 mm

### Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.975, $T_{max}$ = 0.985
7933 measured reflections
2115 independent reflections
1389 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.040

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.121
$S$ = 1.01
2115 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 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···$A$ |
|---|---|---|---|---|
| N2—H2$A$···O3$^{i}$ | 0.86 | 2.15 | 2.970 (2) | 159 |
| N3—H3$A$···O3$^{ii}$ | 0.86 | 2.19 | 3.044 (2) | 170 |
| N3—H3$B$···N1 | 0.86 | 2.30 | 2.657 (2) | 105 |
| N3—H3$B$···O1$^{iii}$ | 0.86 | 2.59 | 3.019 (2) | 112 |
| N3—H3$B$···O2$^{iii}$ | 0.86 | 2.30 | 3.119 (2) | 160 |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors acknowledge the provision of funds for the purchase of a diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha. Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6783).

## References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555-1573.
Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Fun, H.-K., Yeap, C. S., Malladi, S. & Isloor, A. M. (2011). *Acta Cryst.* E**67**, o1786.
Liang, Z.-P., Li, J., Wang, H.-L. & Wang, H.-Q. (2007). *Acta Cryst.* E**63**, o2939.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

*Acta Cryst.* (2012). E**68**, o1724 [doi:10.1107/S1600536812020739]

## 2-[(*E*)-3,4-Dimethoxybenzylidene]hydrazinecarboxamide

**M. Nawaz Tahir, M. Naveed Umar, Akbar Ali and Hazoor Ahmad Shad**

### S1. Comment

The title compound (I), (Fig. 1) has been synthesized as a derivative.

The crystal structures of (*E*)-1-(4-methoxybenzylidene)semicarbazide (Liang *et al.*, 2007) and (*E*)-2-(4-hydroxy-3-methoxybenzylidene) hydrazinecarboxamide (Fun *et al.*, 2011) have been published which are related to the title compound (I).

In (I), the parts of 3,4-dimethoxybenzaldehyde and hydrazinecarboxamide A (C1—C9/O1/O2) and B (N1/N2/C10/N3/O3), are almost planar with r. m. s. deviation of 0.0770 and 0.0159 Å, respectively. The dihedral angle between A/B is 53.82 (6)°. There exist intramolecular H–bonding of N—H···N type (Table 1, Fig. 1) and form *S*(5) ring motif (Bernstein *et al.*, 1995). Each molecule is interlinked with three molecules due to H-bondings of N—H···O type. There exist $R_1^2(5)$, $R_2^2(8)$ and $R_2^4(14)$ ring motifs (Table 1, Fig. 2). The molecules are interliked in the form of two-dimensional polymeric sheets in the plane $(\bar{2}01)$ and with base vectors [100] and [102].

### S2. Experimental

Equimolar quantities of 3,4-dimethoxybenzaldehyde and hydrazinecarboxamide were refluxed in methanol for 45 min resulting in yellow solution. The solution was kept at room temperature which affoarded yellow prisms after 48 h.

### S3. Refinement

The H-atoms were positioned geometrically (C–H = 0.93–0.96 Å and N—H = 0.86 Å) and refined as riding with $U_{iso}$(H) = x$U_{eq}$(C. N), where x = 1.5 for methyl and x = 1.2 for all other H-atoms.

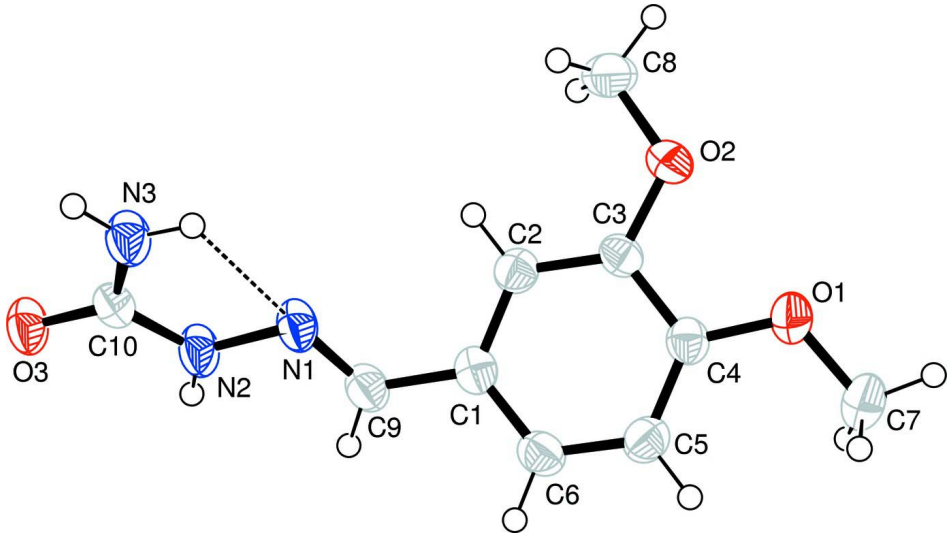



**Figure 1**

View of the title compound with displacement ellipsoids drawn at the 50% probability level. The dotted lines indicate the intra-molecular hydrogen bond.

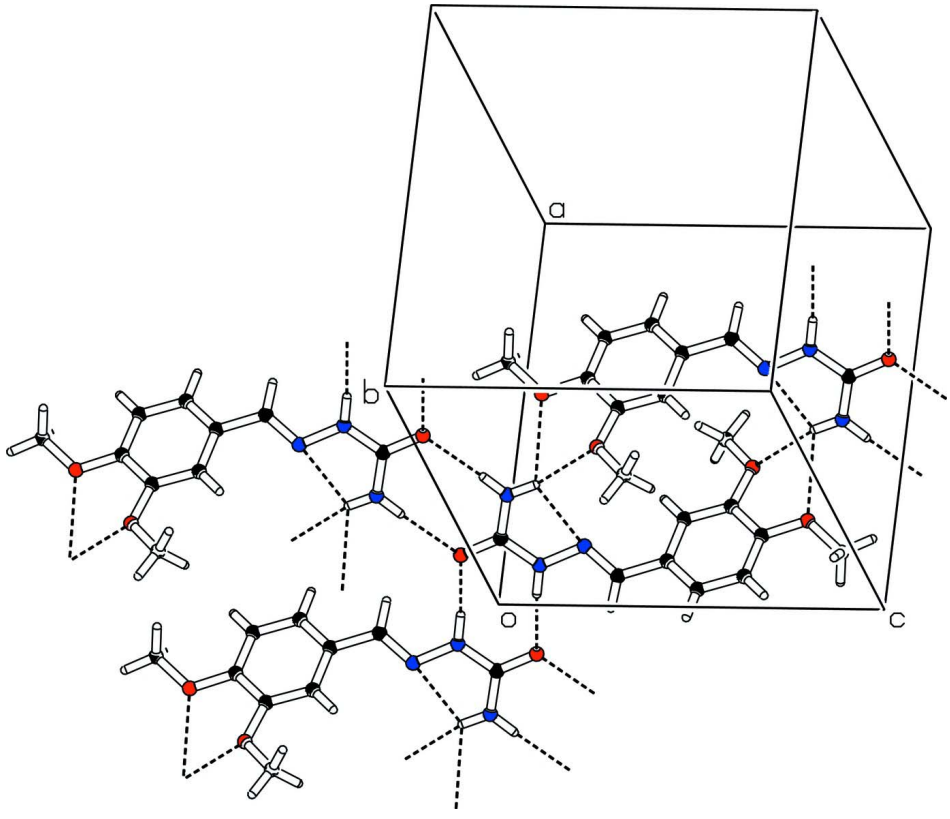



**Figure 2**

Partial packnig diagram showing molecules interlinked to form polymeric sheets with various ring motifs.

**2-[(*E*)-3,4-Dimethoxybenzylidene]hydrazinecarboxamide**

*Crystal data*

$C_{10}H_{13}N_3O_3$
$M_r = 223.23$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 22.2300$ (7) Å
$b = 7.6367$ (3) Å
$c = 15.6482$ (6) Å
$\beta = 126.234$ (1)°
$V = 2142.76$ (14) Å$^3$
$Z = 8$
$F(000) = 944$
$D_x = 1.384$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1389 reflections
$\theta = 2.3$–26.0°
$\mu = 0.10$ mm$^{-1}$
$T = 296$ K
Prism, yellow
0.25 × 0.18 × 0.15 mm

*Data collection*

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.00 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.975$, $T_{max} = 0.985$
7933 measured reflections
2115 independent reflections
1389 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.3°$
$h = -26 \rightarrow 27$
$k = -9 \rightarrow 9$
$l = -19 \rightarrow 19$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.121$
$S = 1.01$
2115 reflections
147 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
$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.0283P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.30582 (7) | −0.13305 (19) | 0.72476 (10) | 0.0464 (5) |
| O2 | 0.34785 (7) | 0.03543 (19) | 0.62646 (10) | 0.0454 (5) |
| O3 | −0.01303 (7) | 0.24827 (17) | −0.02345 (10) | 0.0447 (5) |
| N1 | 0.10093 (8) | 0.0967 (2) | 0.23838 (12) | 0.0404 (5) |
| N2 | 0.04892 (8) | 0.0982 (2) | 0.12929 (12) | 0.0421 (5) |

| | | | | |
|---|---|---|---|---|
| N3 | 0.06302 (9) | 0.3952 (2) | 0.13090 (13) | 0.0445 (6) |
| C1 | 0.15297 (10) | −0.0591 (2) | 0.40077 (15) | 0.0347 (6) |
| C2 | 0.22548 (10) | 0.0085 (2) | 0.45703 (15) | 0.0356 (6) |
| C3 | 0.27503 (10) | −0.0179 (2) | 0.56479 (14) | 0.0331 (6) |
| C4 | 0.25221 (10) | −0.1097 (2) | 0.61917 (15) | 0.0345 (6) |
| C5 | 0.18023 (10) | −0.1725 (3) | 0.56366 (15) | 0.0394 (7) |
| C6 | 0.13153 (10) | −0.1495 (3) | 0.45538 (15) | 0.0397 (7) |
| C7 | 0.28984 (13) | −0.2490 (3) | 0.78007 (16) | 0.0527 (8) |
| C8 | 0.37782 (11) | 0.1099 (3) | 0.57544 (17) | 0.0490 (8) |
| C9 | 0.10014 (10) | −0.0369 (3) | 0.28603 (15) | 0.0400 (7) |
| C10 | 0.03155 (9) | 0.2502 (3) | 0.07501 (15) | 0.0347 (6) |
| H2 | 0.24028 | 0.07157 | 0.42152 | 0.0427* |
| H2A | 0.02764 | 0.00235 | 0.09601 | 0.0505* |
| H3A | 0.05296 | 0.49443 | 0.09922 | 0.0534* |
| H3B | 0.09351 | 0.38993 | 0.19885 | 0.0534* |
| H5 | 0.16442 | −0.23070 | 0.59936 | 0.0473* |
| H6 | 0.08359 | −0.19543 | 0.41857 | 0.0476* |
| H7A | 0.24962 | −0.20238 | 0.77979 | 0.0791* |
| H7B | 0.33332 | −0.26156 | 0.85185 | 0.0791* |
| H7C | 0.27580 | −0.36131 | 0.74591 | 0.0791* |
| H8A | 0.35195 | 0.21675 | 0.54077 | 0.0735* |
| H8B | 0.37181 | 0.02896 | 0.52397 | 0.0735* |
| H8C | 0.42987 | 0.13423 | 0.62723 | 0.0735* |
| H9 | 0.06472 | −0.12360 | 0.24658 | 0.0479* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0482 (8) | 0.0532 (9) | 0.0289 (8) | −0.0080 (7) | 0.0179 (7) | 0.0017 (7) |
| O2 | 0.0369 (8) | 0.0576 (9) | 0.0338 (8) | −0.0152 (7) | 0.0165 (7) | −0.0050 (7) |
| O3 | 0.0471 (8) | 0.0416 (9) | 0.0264 (7) | 0.0006 (6) | 0.0113 (7) | −0.0004 (6) |
| N1 | 0.0408 (9) | 0.0384 (10) | 0.0272 (9) | 0.0008 (7) | 0.0120 (8) | −0.0009 (7) |
| N2 | 0.0448 (10) | 0.0355 (9) | 0.0263 (9) | −0.0042 (8) | 0.0102 (8) | −0.0018 (7) |
| N3 | 0.0476 (10) | 0.0351 (10) | 0.0320 (10) | −0.0030 (8) | 0.0132 (8) | −0.0010 (7) |
| C1 | 0.0362 (10) | 0.0307 (10) | 0.0330 (11) | 0.0031 (8) | 0.0181 (9) | −0.0009 (8) |
| C2 | 0.0401 (11) | 0.0327 (11) | 0.0345 (11) | −0.0023 (8) | 0.0224 (9) | −0.0013 (8) |
| C3 | 0.0321 (10) | 0.0330 (10) | 0.0303 (11) | −0.0042 (8) | 0.0163 (9) | −0.0062 (8) |
| C4 | 0.0374 (10) | 0.0335 (10) | 0.0304 (10) | 0.0001 (8) | 0.0188 (9) | −0.0022 (8) |
| C5 | 0.0417 (11) | 0.0418 (12) | 0.0394 (12) | −0.0001 (9) | 0.0266 (10) | 0.0031 (9) |
| C6 | 0.0307 (10) | 0.0406 (12) | 0.0421 (12) | 0.0005 (9) | 0.0184 (9) | 0.0015 (9) |
| C7 | 0.0604 (14) | 0.0619 (16) | 0.0364 (12) | −0.0022 (11) | 0.0289 (11) | 0.0071 (11) |
| C8 | 0.0462 (12) | 0.0524 (14) | 0.0524 (14) | −0.0126 (10) | 0.0313 (12) | −0.0047 (11) |
| C9 | 0.0350 (11) | 0.0397 (12) | 0.0337 (11) | −0.0015 (9) | 0.0140 (10) | −0.0014 (9) |
| C10 | 0.0289 (10) | 0.0391 (11) | 0.0294 (10) | −0.0001 (8) | 0.0136 (9) | −0.0010 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C4 | 1.362 (2) | C2—C3 | 1.379 (3) |
| O1—C7 | 1.422 (3) | C3—C4 | 1.408 (3) |
| O2—C3 | 1.368 (3) | C4—C5 | 1.379 (3) |
| O2—C8 | 1.426 (3) | C5—C6 | 1.380 (3) |
| O3—C10 | 1.245 (2) | C2—H2 | 0.9300 |
| N1—N2 | 1.385 (2) | C5—H5 | 0.9300 |
| N1—C9 | 1.270 (3) | C6—H6 | 0.9300 |
| N2—C10 | 1.353 (3) | C7—H7A | 0.9600 |
| N3—C10 | 1.326 (3) | C7—H7B | 0.9600 |
| N2—H2A | 0.8600 | C7—H7C | 0.9600 |
| N3—H3A | 0.8600 | C8—H8A | 0.9600 |
| N3—H3B | 0.8600 | C8—H8B | 0.9600 |
| C1—C9 | 1.463 (3) | C8—H8C | 0.9600 |
| C1—C2 | 1.401 (3) | C9—H9 | 0.9300 |
| C1—C6 | 1.384 (3) | | |
| | | | |
| C4—O1—C7 | 117.75 (18) | N2—C10—N3 | 117.33 (17) |
| C3—O2—C8 | 118.26 (15) | O3—C10—N2 | 119.32 (19) |
| N2—N1—C9 | 116.05 (17) | C1—C2—H2 | 120.00 |
| N1—N2—C10 | 120.10 (15) | C3—C2—H2 | 120.00 |
| N1—N2—H2A | 120.00 | C4—C5—H5 | 120.00 |
| C10—N2—H2A | 120.00 | C6—C5—H5 | 120.00 |
| C10—N3—H3A | 120.00 | C1—C6—H6 | 120.00 |
| C10—N3—H3B | 120.00 | C5—C6—H6 | 120.00 |
| H3A—N3—H3B | 120.00 | O1—C7—H7A | 109.00 |
| C2—C1—C6 | 118.95 (18) | O1—C7—H7B | 109.00 |
| C2—C1—C9 | 121.3 (2) | O1—C7—H7C | 109.00 |
| C6—C1—C9 | 119.7 (2) | H7A—C7—H7B | 109.00 |
| C1—C2—C3 | 120.4 (2) | H7A—C7—H7C | 109.00 |
| C2—C3—C4 | 119.9 (2) | H7B—C7—H7C | 109.00 |
| O2—C3—C4 | 114.88 (16) | O2—C8—H8A | 109.00 |
| O2—C3—C2 | 125.2 (2) | O2—C8—H8B | 109.00 |
| O1—C4—C5 | 125.2 (2) | O2—C8—H8C | 109.00 |
| O1—C4—C3 | 115.4 (2) | H8A—C8—H8B | 109.00 |
| C3—C4—C5 | 119.39 (18) | H8A—C8—H8C | 109.00 |
| C4—C5—C6 | 120.3 (2) | H8B—C8—H8C | 109.00 |
| C1—C6—C5 | 121.0 (2) | N1—C9—H9 | 119.00 |
| N1—C9—C1 | 122.29 (19) | C1—C9—H9 | 119.00 |
| O3—C10—N3 | 123.4 (2) | | |
| | | | |
| C7—O1—C4—C3 | −169.75 (18) | C2—C1—C9—N1 | −31.7 (3) |
| C7—O1—C4—C5 | 8.3 (3) | C6—C1—C9—N1 | 148.8 (2) |
| C8—O2—C3—C2 | −6.2 (3) | C1—C2—C3—O2 | 176.97 (18) |
| C8—O2—C3—C4 | 172.38 (17) | C1—C2—C3—C4 | −1.5 (3) |
| C9—N1—N2—C10 | 162.2 (2) | O2—C3—C4—O1 | −0.4 (2) |
| N2—N1—C9—C1 | 178.4 (2) | O2—C3—C4—C5 | −178.58 (18) |

| | | | |
|---|---|---|---|
| N1—N2—C10—O3 | 177.1 (2) | C2—C3—C4—O1 | 178.23 (17) |
| N1—N2—C10—N3 | −3.6 (3) | C2—C3—C4—C5 | 0.1 (3) |
| C6—C1—C2—C3 | 1.3 (3) | O1—C4—C5—C6 | −176.4 (2) |
| C9—C1—C2—C3 | −178.29 (19) | C3—C4—C5—C6 | 1.6 (3) |
| C2—C1—C6—C5 | 0.4 (3) | C4—C5—C6—C1 | −1.9 (3) |
| C9—C1—C6—C5 | 180.0 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···O3$^{i}$ | 0.86 | 2.15 | 2.970 (2) | 159 |
| N3—H3*A*···O3$^{ii}$ | 0.86 | 2.19 | 3.044 (2) | 170 |
| N3—H3*B*···N1 | 0.86 | 2.30 | 2.657 (2) | 105 |
| N3—H3*B*···O1$^{iii}$ | 0.86 | 2.59 | 3.019 (2) | 112 |
| N3—H3*B*···O2$^{iii}$ | 0.86 | 2.30 | 3.119 (2) | 160 |

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*, −*y*+1, −*z*; (iii) −*x*+1/2, −*y*+1/2, −*z*+1.