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# 1-Phenyl-1*H*-pyrazole-4-carbaldehyde

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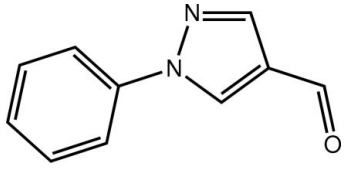
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.050; $wR$ factor = 0.136; data-to-parameter ratio = 12.0.

In the title molecule, $C_{10}H_8N_2O$, the five- and six-membered rings form a dihedral angle of 10.14 (9)°. The aldehyde group is almost coplanar with the pyrazole ring to which it is connected [O—C—C—C torsion angle = −179.35 (17)°]. In the crystal, inversion dimers are linked by four C—H···O interactions as the carbonyl O atom accepts two such bonds. The dimeric aggregates are linked into supramolecular layers in the *ac* plane by C—H···$\pi$ and $\pi$–$\pi$ [ring centroid(pyrrole)···ring centroid(phenyl) = 3.8058 (10) Å] interactions.

## Related literature

For the anti-bacterial properties of pyrazole derivatives, see: Kane *et al.* (2003). For related structures, see: Asiri, Al-Youbi, *et al.* (2012); Asiri, Faidallah *et al.* (2012). For the synthesis, see: Vera-DiVaio *et al.* (2009).



## Experimental

### *Crystal data*

$C_{10}H_8N_2O$
$M_r$ = 172.18
Monoclinic, $P2_1/n$
$a$ = 11.1657 (10) Å
$b$ = 5.0858 (4) Å
$c$ = 15.3034 (11) Å
$\beta$ = 111.130 (9)°
$V$ = 810.60 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 100 K
0.30 × 0.30 × 0.15 mm

### *Data collection*

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min}$ = 0.972, $T_{max}$ = 0.986
3485 measured reflections
1814 independent reflections
1359 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.050

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.136
$S$ = 0.99
1814 reflections
151 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

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

*Cg*1 is the centroid of the C5–C10 ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C4—H4···O1$^{i}$ | 0.960 (19) | 2.432 (19) | 3.379 (2) | 168.6 (13) |
| C10—H10···O1$^{i}$ | 0.978 (19) | 2.335 (19) | 3.303 (2) | 170.8 (16) |
| C8—H8···*Cg*1$^{ii}$ | 0.978 (18) | 2.947 (18) | 3.761 (2) | 141.4 (14) |

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5843).

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# supplementary materials

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## 1-Phenyl-1*H*-pyrazole-4-carbaldehyde

**Abdullah M. Asiri, Hassan M. Faidallah, Tariq R. Sobahi, Seik Weng Ng and Edward R. T. Tiekink**

### Comment

In continuation of structural studies of pyrazole derivatives (Asiri, Al-Youbi, *et al.*, 2012; Asiri, Faidallah *et al.*, 2012), of interest, for example, owing to their anti-bacterial activity (Kane *et al.*, 2003), the title compound, 1-phenyl-1*H*-pyrazole-4-carbaldehyde (I), was investigated crystallographically.

In (I), Fig. 1, the dihedral angle between the five- and six-membered rings is 10.14 (9) °, indicating a slight twist in the molecule. The aldehyde group is co-planar with the pyrazole ring to which it is connected as seen in the value of the O1—C1—C2—C3 torsion angle of -179.35 (17)°.

Inversion related molecules are connected into dimers *via* C—H···O interactions involving a bifurcated carbonyl-O atom, Table 1. Dimers are linked into supramolecular layers in the *ac* plane *via* C—H···$\pi$ and $\pi$—$\pi$ interactions occurring between the five- and six-membered rings [ring centroid···ring centroid distance = 3.8058 (10) Å, angle of inclination = 10.14 (9)° for symmetry operation: *x*, -1 + *y*, *z*], Fig. 2 and Table 1. Layers stack with no specific intermolecular interactions between them, Fig. 3.

### Experimental

*N*,*N*-Dimethylformamide (25.6 ml, 0.33 mmol) was stirred in around flask within an ice-bath and $POCl_3$ (21.6 ml, 0.23 mmol) was added drop-wise. Then *N*-phenylpyrazole was added (4.4 ml, 0.033 mmol) to this cold mixture. The reaction was allowed to warm to room temperature and then heated at reflux for 6 h. The temperature was kept at 368–373 K. When the reaction was completed, the contents were poured onto crushed ice and made weakly alkaline with a saturated solution of sodium carbonate. The solid was filtered off, washed with water and recrystallized from ethanol. Yield: 65%. *M*.pt. 358–359 K. (lit. 358 K; Vera-DiVaio *et al.*, 2009).

### Refinement

H-atoms were freely refined; the range of C—H bond lengths is 0.960 (17)–1.023 (18) Å. Owing to poor agreement, the ($\bar{3}$ 2 6) reflection was omitted from the final cycles of refinement.

### Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

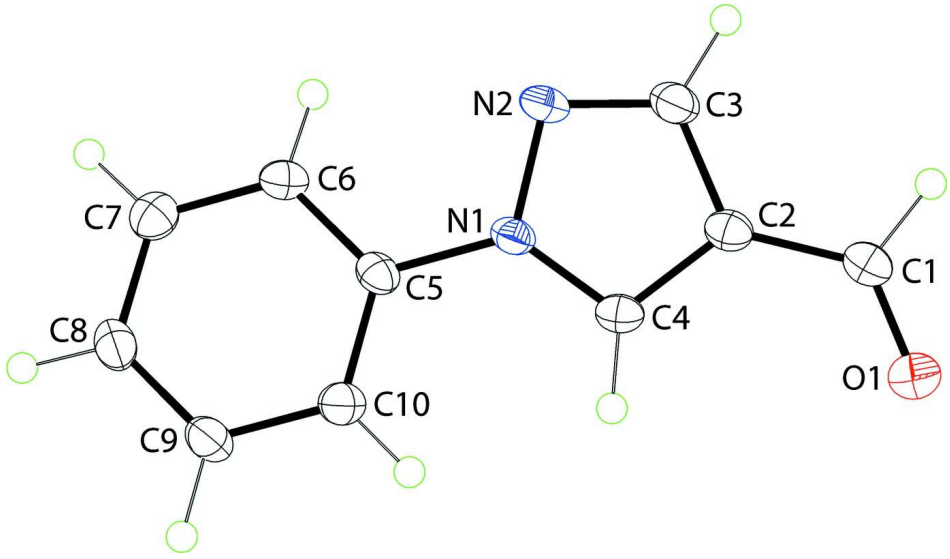


**Figure 1**

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

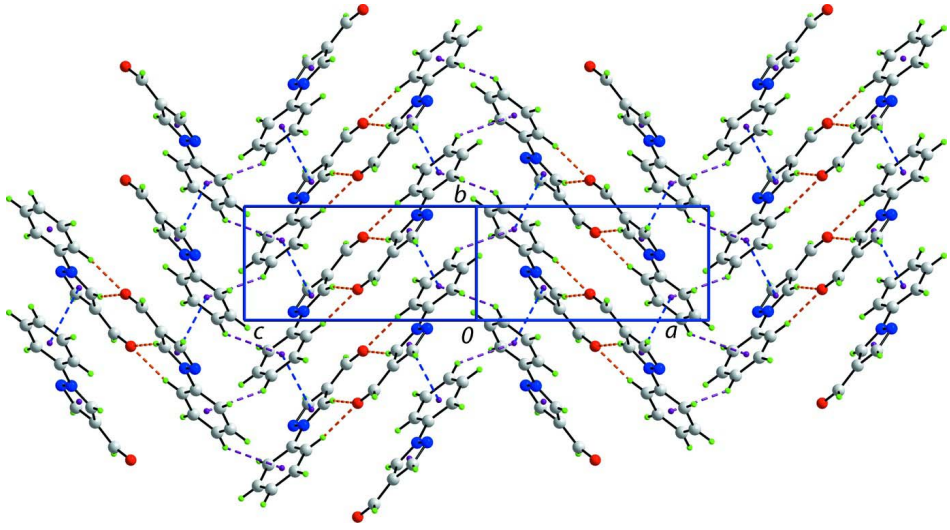


**Figure 2**

A view of the supramolecular layer in the *ac* plane in (I). The C—H···O, C—H···$\pi$ and $\pi$—$\pi$ interactions are shown as orange, purple and blue dashed lines, respectively.

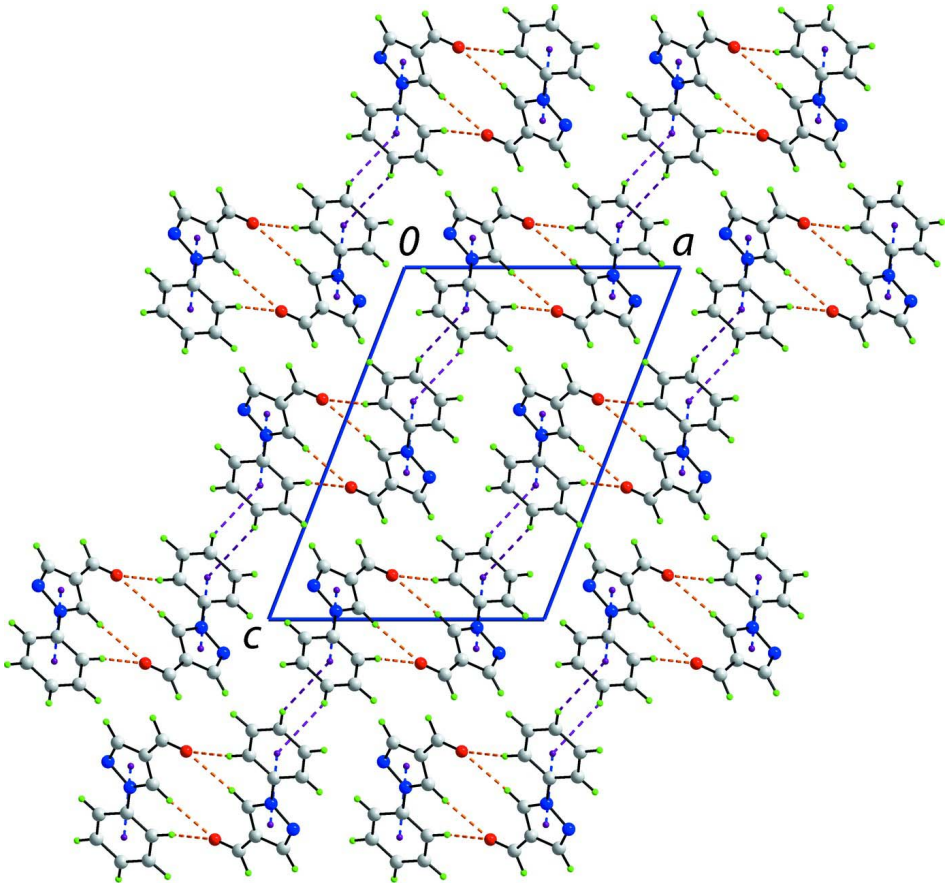


**Figure 3**

A view in projection down the *b* axis of the unit-cell contents of (I). The C—H···O, C—H···$\pi$ and $\pi$—$\pi$ interactions are shown as orange, purple and blue dashed lines, respectively.

### 1-Phenyl-1*H*-pyrazole-4-carbaldehyde

*Crystal data*

$C_{10}H_8N_2O$
$M_r$ = 172.18
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a$ = 11.1657 (10) Å
$b$ = 5.0858 (4) Å
$c$ = 15.3034 (11) Å
$\beta$ = 111.130 (9)°
$V$ = 810.60 (11) Å$^3$
$Z$ = 4
$F(000)$ = 360
$D_x$ = 1.411 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 1251 reflections
$\theta$ = 2.8–27.5°
$\mu$ = 0.10 mm$^{-1}$
$T$ = 100 K
Prism, light-brown
0.30 × 0.30 × 0.15 mm

*Data collection*

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm$^{-1}$
$\omega$ scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min}$ = 0.972, $T_{max}$ = 0.986
3485 measured reflections
1814 independent reflections

1359 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.8°$
$h = -14 \rightarrow 14$
$k = -5 \rightarrow 6$
$l = -19 \rightarrow 13$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.136$
$S = 0.99$
1814 reflections
151 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0732P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.013 (4)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.11384 (11) | 0.2706 (2) | 0.62516 (8) | 0.0286 (4) |
| N1 | 0.26714 (12) | 0.9228 (3) | 0.52304 (9) | 0.0196 (3) |
| N2 | 0.38346 (13) | 0.9268 (3) | 0.59584 (9) | 0.0247 (4) |
| C1 | 0.21644 (15) | 0.3842 (3) | 0.65682 (11) | 0.0230 (4) |
| C2 | 0.25665 (15) | 0.6026 (3) | 0.61478 (11) | 0.0209 (4) |
| C3 | 0.37465 (16) | 0.7329 (3) | 0.65005 (12) | 0.0245 (4) |
| C4 | 0.18990 (15) | 0.7336 (3) | 0.53228 (11) | 0.0204 (4) |
| C5 | 0.24267 (15) | 1.1131 (3) | 0.45059 (10) | 0.0196 (4) |
| C6 | 0.34207 (16) | 1.2698 (3) | 0.44624 (11) | 0.0226 (4) |
| C7 | 0.31714 (17) | 1.4569 (4) | 0.37669 (12) | 0.0248 (4) |
| C8 | 0.19463 (17) | 1.4863 (4) | 0.31105 (11) | 0.0247 (4) |
| C9 | 0.09706 (17) | 1.3272 (4) | 0.31553 (12) | 0.0263 (4) |
| C10 | 0.11990 (16) | 1.1412 (3) | 0.38573 (12) | 0.0242 (4) |
| H1 | 0.2818 (16) | 0.327 (4) | 0.7201 (13) | 0.022 (4)* |
| H3 | 0.4456 (17) | 0.689 (4) | 0.7089 (13) | 0.027 (5)* |
| H4 | 0.1060 (17) | 0.707 (3) | 0.4858 (12) | 0.022 (5)* |
| H6 | 0.4267 (19) | 1.239 (4) | 0.4903 (14) | 0.033 (5)* |
| H7 | 0.3846 (16) | 1.567 (4) | 0.3715 (12) | 0.023 (5)* |
| H8 | 0.1801 (15) | 1.619 (4) | 0.2621 (12) | 0.019 (4)* |
| H9 | 0.0057 (18) | 1.351 (4) | 0.2696 (14) | 0.037 (5)* |
| H10 | 0.0500 (15) | 1.030 (4) | 0.3886 (12) | 0.023 (5)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0250 (7) | 0.0306 (8) | 0.0274 (6) | −0.0045 (5) | 0.0063 (5) | −0.0006 (6) |
| N1 | 0.0183 (7) | 0.0214 (8) | 0.0154 (6) | 0.0002 (5) | 0.0015 (5) | −0.0005 (6) |
| N2 | 0.0193 (7) | 0.0286 (8) | 0.0194 (7) | −0.0011 (6) | −0.0013 (6) | 0.0005 (6) |
| C1 | 0.0229 (9) | 0.0254 (9) | 0.0184 (8) | 0.0017 (7) | 0.0046 (7) | −0.0011 (7) |
| C2 | 0.0205 (8) | 0.0216 (9) | 0.0186 (7) | 0.0007 (6) | 0.0047 (6) | −0.0032 (7) |
| C3 | 0.0225 (9) | 0.0270 (10) | 0.0192 (8) | 0.0005 (7) | 0.0018 (7) | 0.0001 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C4 | 0.0182 (8) | 0.0220 (9) | 0.0185 (7) | −0.0007 (6) | 0.0035 (7) | −0.0038 (7) |
| C5 | 0.0230 (8) | 0.0197 (9) | 0.0142 (7) | 0.0011 (6) | 0.0042 (6) | −0.0017 (6) |
| C6 | 0.0196 (9) | 0.0263 (10) | 0.0191 (8) | −0.0015 (7) | 0.0038 (7) | −0.0026 (7) |
| C7 | 0.0271 (9) | 0.0243 (9) | 0.0238 (8) | −0.0024 (7) | 0.0103 (7) | −0.0034 (8) |
| C8 | 0.0313 (9) | 0.0205 (9) | 0.0209 (8) | 0.0021 (7) | 0.0076 (7) | 0.0027 (8) |
| C9 | 0.0242 (9) | 0.0253 (10) | 0.0236 (8) | 0.0031 (7) | 0.0017 (7) | 0.0037 (8) |
| C10 | 0.0212 (9) | 0.0222 (9) | 0.0251 (8) | −0.0010 (7) | 0.0034 (7) | 0.0002 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.2168 (19) | C5—C10 | 1.380 (2) |
| N1—C4 | 1.333 (2) | C5—C6 | 1.387 (2) |
| N1—N2 | 1.3731 (17) | C6—C7 | 1.379 (2) |
| N1—C5 | 1.422 (2) | C6—H6 | 0.96 (2) |
| N2—C3 | 1.315 (2) | C7—C8 | 1.383 (2) |
| C1—C2 | 1.435 (2) | C7—H7 | 0.963 (18) |
| C1—H1 | 1.023 (18) | C8—C9 | 1.379 (3) |
| C2—C4 | 1.384 (2) | C8—H8 | 0.978 (18) |
| C2—C3 | 1.398 (2) | C9—C10 | 1.384 (2) |
| C3—H3 | 0.986 (18) | C9—H9 | 1.016 (18) |
| C4—H4 | 0.960 (17) | C10—H10 | 0.977 (17) |
| | | | |
| C4—N1—N2 | 112.57 (12) | C10—C5—N1 | 119.55 (14) |
| C4—N1—C5 | 128.61 (13) | C6—C5—N1 | 119.75 (14) |
| N2—N1—C5 | 118.81 (13) | C7—C6—C5 | 119.40 (15) |
| C3—N2—N1 | 103.68 (13) | C7—C6—H6 | 122.1 (12) |
| O1—C1—C2 | 126.22 (15) | C5—C6—H6 | 118.4 (12) |
| O1—C1—H1 | 119.3 (10) | C6—C7—C8 | 120.47 (16) |
| C2—C1—H1 | 114.5 (10) | C6—C7—H7 | 121.1 (11) |
| C4—C2—C3 | 104.28 (15) | C8—C7—H7 | 118.4 (11) |
| C4—C2—C1 | 129.01 (15) | C9—C8—C7 | 119.52 (17) |
| C3—C2—C1 | 126.71 (15) | C9—C8—H8 | 121.9 (10) |
| N2—C3—C2 | 112.73 (14) | C7—C8—H8 | 118.6 (10) |
| N2—C3—H3 | 121.7 (11) | C8—C9—C10 | 120.80 (16) |
| C2—C3—H3 | 125.6 (11) | C8—C9—H9 | 120.6 (11) |
| N1—C4—C2 | 106.74 (14) | C10—C9—H9 | 118.5 (11) |
| N1—C4—H4 | 121.3 (10) | C5—C10—C9 | 119.10 (16) |
| C2—C4—H4 | 131.9 (11) | C5—C10—H10 | 120.7 (10) |
| C10—C5—C6 | 120.70 (15) | C9—C10—H10 | 120.2 (10) |
| | | | |
| C4—N1—N2—C3 | 0.04 (18) | N2—N1—C5—C10 | −169.04 (14) |
| C5—N1—N2—C3 | 179.12 (14) | C4—N1—C5—C6 | −170.62 (15) |
| O1—C1—C2—C4 | 1.2 (3) | N2—N1—C5—C6 | 10.5 (2) |
| O1—C1—C2—C3 | −179.35 (17) | C10—C5—C6—C7 | 0.6 (2) |
| N1—N2—C3—C2 | 0.16 (19) | N1—C5—C6—C7 | −178.94 (14) |
| C4—C2—C3—N2 | −0.3 (2) | C5—C6—C7—C8 | −0.8 (3) |
| C1—C2—C3—N2 | −179.83 (16) | C6—C7—C8—C9 | 0.0 (3) |
| N2—N1—C4—C2 | −0.21 (18) | C7—C8—C9—C10 | 1.0 (3) |
| C5—N1—C4—C2 | −179.18 (15) | C6—C5—C10—C9 | 0.4 (3) |
| C3—C2—C4—N1 | 0.29 (17) | N1—C5—C10—C9 | 179.95 (15) |

| | | | |
|---|---|---|---|
| C1—C2—C4—N1 | 179.81 (16) | C8—C9—C10—C5 | −1.2 (3) |
| C4—N1—C5—C10 | 9.9 (3) | | |

*Hydrogen-bond geometry (Å, º)*

Cg1 is the centroid of the C5–C10 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4···O1$^{i}$ | 0.960 (19) | 2.432 (19) | 3.379 (2) | 168.6 (13) |
| C10—H10···O1$^{i}$ | 0.978 (19) | 2.335 (19) | 3.303 (2) | 170.8 (16) |
| C8—H8···*Cg*1$^{ii}$ | 0.978 (18) | 2.947 (18) | 3.761 (2) | 141.4 (14) |

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