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1-[(3,5-Dimethyl-1*H*-pyrazol-1-yl)-carbonyl]-5-methylindolizine-3-carbonitrile

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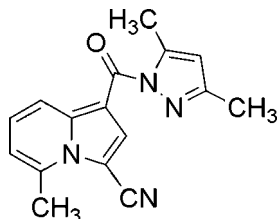
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.135; data-to-parameter ratio = 12.2.

In the title molecule, $\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}$, the indolizine ring system is essentially planar, with a maximum deviation of 0.013 (3) Å, and forms a dihedral angle of 7.52 (12)° with the pyrazole ring. In the crystal, weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and $\pi-\pi$ stacking interactions, with a centroid-centroid distance of 3.6378 (16) Å, link molecules along [001].

Related literature

For biological applications of indolizines and pyrazoles, see: Tukulula *et al.* (2010); James *et al.* (2008); Teklu *et al.* (2005); McDonald *et al.* (2006); Jagerovic *et al.* (2002). For background and the synthesis of related heterocycles, see: Gu *et al.* (2011); Shen *et al.* (2006, 2008); Wang, *et al.* (2000).



Experimental

Crystal data

| | |
|--|-----------------------------------|
| $\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}$ | $V = 1382.4$ (4) Å ³ |
| $M_r = 278.31$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 8.5911$ (18) Å | $\mu = 0.09$ mm ⁻¹ |
| $b = 23.3760$ (15) Å | $T = 291$ K |
| $c = 7.5816$ (12) Å | $0.30 \times 0.25 \times 0.20$ mm |
| $\beta = 114.775$ (3)° | |

Data collection

| | |
|--|--|
| Bruker SMART APEX diffractometer | 7822 measured reflections |
| Absorption correction: multi-scan (SADABS; Bruker, 2000) | 2361 independent reflections |
| $T_{\min} = 0.974$, $T_{\max} = 0.983$ | 1728 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.037$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.048$ | 1 restraint |
| $wR(F^2) = 0.135$ | H-atom parameters constrained |
| $S = 1.05$ | $\Delta\rho_{\text{max}} = 0.19$ e Å ⁻³ |
| 2361 reflections | $\Delta\rho_{\text{min}} = -0.15$ e Å ⁻³ |
| 194 parameters | |

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

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C10}-\text{H10B}\cdots\text{O1}^i$ | 0.96 | 2.56 | 3.435 (4) | 151 |

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL and PLATON (Spek, 2009); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5554).

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supplementary materials

Acta Cryst. (2012). E68, o3399 [doi:10.1107/S1600536812046727]

1-[(3,5-Dimethyl-1*H*-pyrazol-1-yl)carbonyl]-5-methylindolizine-3-carbonitrile

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Comment

Indolizines and pyrazoles are important classes of bio-active drug targets in the pharmaceutical industry, as they are the core structure of numerous biologically active compounds (Tukulula *et al.*, 2010; James *et al.*, 2008; Teklu *et al.*, 2005; McDonald *et al.*, 2006; Jagerovic *et al.*, 2002). In our continuing studies on the synthesis and properties of heterocycles (Gu *et al.*, 2011; Shen *et al.*, 2008; Shen *et al.*, 2006; Wang, *et al.*, 2000) we have prepared (Fig. 1) and determined the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 2. The indolizine ring system is essentially planar with a maximum deviation for atom C3 of 0.013 (3)Å. The dihedral angle formed by the indolizine ring system and the pyrazole ring is 7.52 (12)°. In the crystal, weak C—H···O hydrogen bonds and π – π stacking interactions, with $\text{Cg1}\cdots\text{Cg2}^i = 3.6378$ (16)Å, link molecules along [001] (Fig. 3). Cg1 and Cg2 are the centroids of the N1/C5–C8 and N1/C1–C5 rings (symmetry code (i): $x, 1/2-y, -1/2+z$).

Experimental

Methyl-3-cyano-5-methylindolizine-1-carboxylate was prepared through 1,3-dipolar cycloaddition according to a procedure described in the literature (Wang, *et al.*, 2000). A suspension of N-cyano-2-methylpyridinium bromide ($\text{C}_5\text{H}_4\text{N}^+\text{CH}_3\text{CN}\cdot\text{Br}^-$) (10 mmol), ethyl acrylate (40 mmol), Et_3N (20 ml) and CrO_3 (20 mmol) in DMF (40 ml) was stirred at 363K for 4 h (monitored by TLC). The mixture was cooled to room temperature and poured into 5% aqueous HCl (150 mL). The brown powder was collected by filtration and washed with ethanol (25 mL). After drying the solid was collected 1.28 g (60%).

Methyl-3-cyano-5-methylindolizine-1-carboxylate (5 mmol) was dissolved in 6 ml of ethanol and 20 ml 80% $\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$ (30 mmol) was added dropwise. The solution was refluxed for 6 h and cooled to yield the product, 0.87 g (81%) as 3-cyano-5-methylindolizine-1-carbohydrazide.

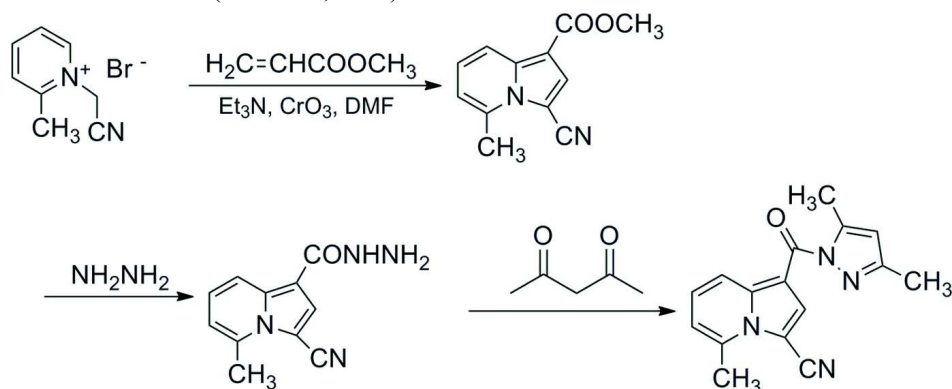
3-cyano-5-methylindolizine-1-carbohydrazide (1 mmol) was dissolved in 2 ml acetic acid, then acetylacetone (2 mmol, dissolved in 2ml ethanol) was added. After stirring for 2 h, the mixture was purified by chromatography [silica gel, 20% ethyl acetate in petroleum ether (60 C90)] to yield colorless block crystals of the title compound, 0.22 g (79%). ¹H-NMR (CDCl_3 , 400 MHz): 2.33 (s, 3H, –CH₃), 2.65 (s, 3H, –CH₃), 3.06 (s, 3H, –CH₃), 6.06 (s, 1H, pyrazole =CH), 6.80 (d, 1H, indolizine =CH), 7.33 (t, 1H, indolizine =CH), 8.52 (d, 1H, indolizine =CH), 8.59 (s, 1H, indolizine =CH).

Refinement

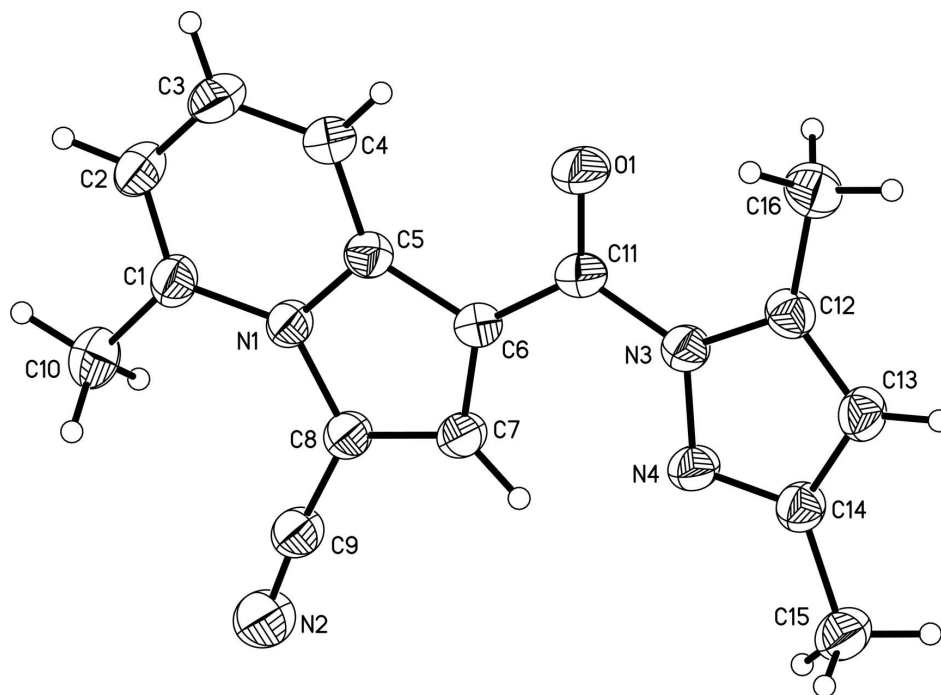
H atoms were placed in calculated positions and allowed to ride, with C—H = 0.93 and 0.96Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.2U_{\text{eq}}(\text{C}_{\text{methyl}})$. The distance for C8—C9 was restrained with the command DFIX 1.4 0.01 C8 C9 in SHELXL (Sheldrick, 2008).

Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

**Figure 1**

The reaction scheme.

**Figure 2**

The molecular structure of title compound. Displacement ellipsoids at the 50% probability level.

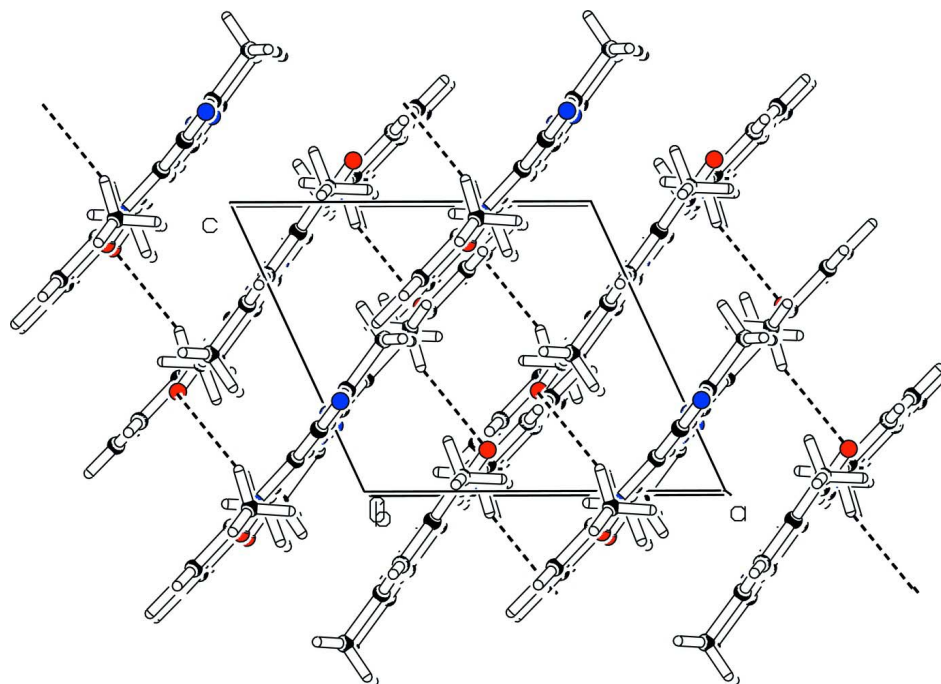


Figure 3

Part of the crystal structure with weak hydrogen bonds shown as dashed lines.

1-[(3,5-Dimethyl-1H-pyrazol-1-yl)carbonyl]-5-methylindolizine-3-carbonitrile

Crystal data

$C_{16}H_{14}N_4O$

$M_r = 278.31$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 8.5911(18)\ \text{\AA}$

$b = 23.3760(15)\ \text{\AA}$

$c = 7.5816(12)\ \text{\AA}$

$\beta = 114.775(3)^\circ$

$V = 1382.4(4)\ \text{\AA}^3$

$Z = 4$

$F(000) = 584$

$D_x = 1.337\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1864 reflections

$\theta = 2.1\text{--}23.1^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 291\ \text{K}$

Block, colorless

$0.30 \times 0.25 \times 0.20\ \text{mm}$

Data collection

Bruker SMART APEX
diffractometer

Radiation source: sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2000)

$T_{\min} = 0.974$, $T_{\max} = 0.983$

7822 measured reflections

2361 independent reflections

1728 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.7^\circ$

$h = -10 \rightarrow 10$

$k = -27 \rightarrow 27$

$l = -8 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.135$

$S = 1.05$

2361 reflections

194 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 0.2279P]$

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

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

$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXTL* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: none

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|--------------|-------------|----------------------------------|
| C1 | 0.4078 (3) | 0.16105 (9) | 0.6276 (3) | 0.0539 (6) |
| C2 | 0.5673 (3) | 0.16955 (11) | 0.7688 (3) | 0.0674 (7) |
| H2 | 0.6342 | 0.1380 | 0.8292 | 0.081* |
| C3 | 0.6350 (3) | 0.22472 (11) | 0.8269 (4) | 0.0706 (7) |
| H3 | 0.7458 | 0.2291 | 0.9227 | 0.085* |
| C4 | 0.5393 (3) | 0.27142 (10) | 0.7437 (3) | 0.0610 (6) |
| H4 | 0.5835 | 0.3079 | 0.7834 | 0.073* |
| C5 | 0.3740 (3) | 0.26454 (9) | 0.5980 (3) | 0.0494 (5) |
| C6 | 0.2428 (2) | 0.30329 (9) | 0.4823 (3) | 0.0473 (5) |
| C7 | 0.1052 (3) | 0.27046 (9) | 0.3576 (3) | 0.0502 (5) |
| H7 | 0.0023 | 0.2849 | 0.2655 | 0.060* |
| C8 | 0.1454 (2) | 0.21350 (7) | 0.3920 (3) | 0.0495 (5) |
| C9 | 0.0408 (3) | 0.16786 (8) | 0.2894 (3) | 0.0572 (6) |
| C10 | 0.3329 (3) | 0.10310 (10) | 0.5630 (4) | 0.0698 (7) |
| H10A | 0.4101 | 0.0746 | 0.6440 | 0.105* |
| H10B | 0.3150 | 0.0971 | 0.4305 | 0.105* |
| H10C | 0.2253 | 0.1003 | 0.5727 | 0.105* |
| C11 | 0.2680 (3) | 0.36492 (10) | 0.5125 (3) | 0.0553 (6) |
| C12 | 0.1506 (3) | 0.46295 (9) | 0.3983 (3) | 0.0514 (5) |
| C13 | 0.0074 (3) | 0.48077 (9) | 0.2476 (3) | 0.0565 (6) |
| H13 | -0.0256 | 0.5186 | 0.2140 | 0.068* |
| C14 | -0.0831 (3) | 0.43187 (9) | 0.1502 (3) | 0.0524 (5) |
| C15 | -0.2500 (3) | 0.42892 (10) | -0.0234 (4) | 0.0673 (7) |
| H15A | -0.2309 | 0.4329 | -0.1389 | 0.101* |

| | | | | |
|------|-------------|--------------|------------|------------|
| H15B | -0.3232 | 0.4592 | -0.0178 | 0.101* |
| H15C | -0.3035 | 0.3927 | -0.0256 | 0.101* |
| C16 | 0.2889 (3) | 0.49760 (11) | 0.5473 (4) | 0.0703 (7) |
| H16A | 0.2630 | 0.5375 | 0.5222 | 0.105* |
| H16B | 0.3963 | 0.4894 | 0.5418 | 0.105* |
| H16C | 0.2966 | 0.4882 | 0.6740 | 0.105* |
| N1 | 0.3117 (2) | 0.20945 (7) | 0.5412 (2) | 0.0481 (5) |
| N2 | -0.0540 (3) | 0.13371 (9) | 0.1932 (3) | 0.0792 (7) |
| N3 | 0.1439 (2) | 0.40334 (7) | 0.3880 (3) | 0.0505 (5) |
| N4 | -0.0015 (2) | 0.38486 (7) | 0.2341 (3) | 0.0543 (5) |
| O1 | 0.3936 (2) | 0.38496 (7) | 0.6446 (3) | 0.0837 (6) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0572 (14) | 0.0536 (13) | 0.0514 (13) | 0.0122 (10) | 0.0232 (12) | 0.0077 (10) |
| C2 | 0.0593 (15) | 0.0719 (17) | 0.0605 (16) | 0.0190 (12) | 0.0147 (14) | 0.0098 (12) |
| C3 | 0.0499 (14) | 0.0874 (19) | 0.0578 (16) | 0.0108 (13) | 0.0061 (12) | 0.0002 (13) |
| C4 | 0.0555 (14) | 0.0631 (14) | 0.0525 (14) | 0.0036 (11) | 0.0108 (12) | -0.0049 (11) |
| C5 | 0.0492 (13) | 0.0542 (13) | 0.0434 (13) | 0.0025 (10) | 0.0179 (11) | -0.0012 (9) |
| C6 | 0.0466 (12) | 0.0495 (12) | 0.0415 (12) | 0.0015 (9) | 0.0141 (10) | -0.0008 (9) |
| C7 | 0.0452 (12) | 0.0535 (13) | 0.0452 (12) | 0.0017 (9) | 0.0125 (10) | 0.0023 (10) |
| C8 | 0.0473 (12) | 0.0498 (13) | 0.0480 (13) | 0.0019 (9) | 0.0165 (11) | 0.0023 (9) |
| C9 | 0.0535 (14) | 0.0513 (13) | 0.0578 (15) | -0.0016 (11) | 0.0145 (12) | 0.0037 (11) |
| C10 | 0.0733 (16) | 0.0571 (15) | 0.0756 (18) | 0.0125 (12) | 0.0280 (14) | 0.0122 (12) |
| C11 | 0.0493 (13) | 0.0569 (14) | 0.0483 (13) | -0.0009 (10) | 0.0092 (12) | -0.0052 (10) |
| C12 | 0.0494 (13) | 0.0480 (12) | 0.0565 (13) | -0.0065 (10) | 0.0220 (11) | -0.0069 (10) |
| C13 | 0.0534 (13) | 0.0433 (12) | 0.0677 (15) | 0.0006 (10) | 0.0205 (12) | 0.0015 (10) |
| C14 | 0.0503 (12) | 0.0483 (12) | 0.0548 (14) | 0.0005 (10) | 0.0183 (11) | 0.0023 (10) |
| C15 | 0.0541 (14) | 0.0627 (15) | 0.0688 (16) | 0.0000 (11) | 0.0096 (13) | 0.0040 (12) |
| C16 | 0.0640 (15) | 0.0585 (15) | 0.0795 (18) | -0.0122 (11) | 0.0213 (14) | -0.0131 (12) |
| N1 | 0.0475 (10) | 0.0517 (10) | 0.0441 (11) | 0.0068 (8) | 0.0181 (9) | 0.0030 (8) |
| N2 | 0.0789 (15) | 0.0564 (13) | 0.0822 (16) | -0.0097 (11) | 0.0141 (13) | 0.0027 (11) |
| N3 | 0.0496 (10) | 0.0445 (10) | 0.0497 (11) | -0.0015 (8) | 0.0134 (9) | -0.0016 (8) |
| N4 | 0.0490 (10) | 0.0487 (10) | 0.0510 (11) | -0.0034 (8) | 0.0071 (9) | -0.0007 (8) |
| O1 | 0.0668 (11) | 0.0636 (10) | 0.0771 (12) | -0.0006 (9) | -0.0126 (10) | -0.0156 (9) |

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

| | | | |
|--------|-----------|----------|-----------|
| C1—C2 | 1.354 (3) | C10—H10B | 0.9600 |
| C1—N1 | 1.392 (3) | C10—H10C | 0.9600 |
| C1—C10 | 1.492 (3) | C11—O1 | 1.217 (3) |
| C2—C3 | 1.407 (3) | C11—N3 | 1.411 (3) |
| C2—H2 | 0.9300 | C12—C13 | 1.347 (3) |
| C3—C4 | 1.353 (3) | C12—N3 | 1.395 (3) |
| C3—H3 | 0.9300 | C12—C16 | 1.490 (3) |
| C4—C5 | 1.396 (3) | C13—C14 | 1.405 (3) |
| C4—H4 | 0.9300 | C13—H13 | 0.9300 |
| C5—N1 | 1.392 (3) | C14—N4 | 1.315 (3) |
| C5—C6 | 1.426 (3) | C14—C15 | 1.487 (3) |

| | | | |
|---------------|-------------|---------------|--------------|
| C6—C7 | 1.394 (3) | C15—H15A | 0.9600 |
| C6—C11 | 1.460 (3) | C15—H15B | 0.9600 |
| C7—C8 | 1.373 (3) | C15—H15C | 0.9600 |
| C7—H7 | 0.9300 | C16—H16A | 0.9600 |
| C8—C9 | 1.4017 (10) | C16—H16B | 0.9600 |
| C8—N1 | 1.405 (3) | C16—H16C | 0.9600 |
| C9—N2 | 1.156 (2) | N3—N4 | 1.374 (2) |
| C10—H10A | 0.9600 | | |
| | | | |
| C2—C1—N1 | 117.2 (2) | O1—C11—N3 | 117.8 (2) |
| C2—C1—C10 | 123.2 (2) | O1—C11—C6 | 122.1 (2) |
| N1—C1—C10 | 119.6 (2) | N3—C11—C6 | 120.15 (19) |
| C1—C2—C3 | 122.0 (2) | C13—C12—N3 | 105.07 (18) |
| C1—C2—H2 | 119.0 | C13—C12—C16 | 129.0 (2) |
| C3—C2—H2 | 119.0 | N3—C12—C16 | 125.9 (2) |
| C4—C3—C2 | 120.2 (2) | C12—C13—C14 | 107.56 (19) |
| C4—C3—H3 | 119.9 | C12—C13—H13 | 126.2 |
| C2—C3—H3 | 119.9 | C14—C13—H13 | 126.2 |
| C3—C4—C5 | 119.6 (2) | N4—C14—C13 | 111.09 (19) |
| C3—C4—H4 | 120.2 | N4—C14—C15 | 120.68 (19) |
| C5—C4—H4 | 120.2 | C13—C14—C15 | 128.2 (2) |
| N1—C5—C4 | 118.9 (2) | C14—C15—H15A | 109.5 |
| N1—C5—C6 | 107.16 (18) | C14—C15—H15B | 109.5 |
| C4—C5—C6 | 133.9 (2) | H15A—C15—H15B | 109.5 |
| C7—C6—C5 | 107.14 (19) | C14—C15—H15C | 109.5 |
| C7—C6—C11 | 132.6 (2) | H15A—C15—H15C | 109.5 |
| C5—C6—C11 | 120.22 (19) | H15B—C15—H15C | 109.5 |
| C8—C7—C6 | 109.30 (19) | C12—C16—H16A | 109.5 |
| C8—C7—H7 | 125.3 | C12—C16—H16B | 109.5 |
| C6—C7—H7 | 125.3 | H16A—C16—H16B | 109.5 |
| C7—C8—C9 | 125.51 (19) | C12—C16—H16C | 109.5 |
| C7—C8—N1 | 107.97 (16) | H16A—C16—H16C | 109.5 |
| C9—C8—N1 | 126.47 (18) | H16B—C16—H16C | 109.5 |
| N2—C9—C8 | 174.1 (2) | C1—N1—C5 | 122.08 (18) |
| C1—C10—H10A | 109.5 | C1—N1—C8 | 129.49 (17) |
| C1—C10—H10B | 109.5 | C5—N1—C8 | 108.43 (16) |
| H10A—C10—H10B | 109.5 | N4—N3—C12 | 111.28 (17) |
| C1—C10—H10C | 109.5 | N4—N3—C11 | 122.09 (16) |
| H10A—C10—H10C | 109.5 | C12—N3—C11 | 126.62 (18) |
| H10B—C10—H10C | 109.5 | C14—N4—N3 | 105.00 (16) |
| | | | |
| N1—C1—C2—C3 | -0.1 (3) | C10—C1—N1—C5 | 178.91 (19) |
| C10—C1—C2—C3 | 179.9 (2) | C2—C1—N1—C8 | 179.34 (19) |
| C1—C2—C3—C4 | 1.1 (4) | C10—C1—N1—C8 | -0.7 (3) |
| C2—C3—C4—C5 | -0.9 (4) | C4—C5—N1—C1 | 1.2 (3) |
| C3—C4—C5—N1 | -0.2 (3) | C6—C5—N1—C1 | -179.24 (16) |
| C3—C4—C5—C6 | -179.6 (2) | C4—C5—N1—C8 | -179.07 (17) |
| N1—C5—C6—C7 | -0.5 (2) | C6—C5—N1—C8 | 0.5 (2) |
| C4—C5—C6—C7 | 178.9 (2) | C7—C8—N1—C1 | 179.42 (18) |

| | | | |
|-----------------|--------------|----------------|--------------|
| N1—C5—C6—C11 | 178.04 (17) | C9—C8—N1—C1 | -3.2 (3) |
| C4—C5—C6—C11 | -2.5 (3) | C7—C8—N1—C5 | -0.2 (2) |
| C5—C6—C7—C8 | 0.4 (2) | C9—C8—N1—C5 | 177.15 (19) |
| C11—C6—C7—C8 | -177.9 (2) | C13—C12—N3—N4 | -0.3 (2) |
| C6—C7—C8—C9 | -177.50 (19) | C16—C12—N3—N4 | 179.45 (18) |
| C6—C7—C8—N1 | -0.1 (2) | C13—C12—N3—C11 | 178.35 (18) |
| C7—C6—C11—O1 | 172.3 (2) | C16—C12—N3—C11 | -1.9 (3) |
| C5—C6—C11—O1 | -5.8 (3) | O1—C11—N3—N4 | 179.5 (2) |
| C7—C6—C11—N3 | -6.9 (3) | C6—C11—N3—N4 | -1.2 (3) |
| C5—C6—C11—N3 | 175.01 (17) | O1—C11—N3—C12 | 1.0 (3) |
| N3—C12—C13—C14 | 0.3 (2) | C6—C11—N3—C12 | -179.75 (18) |
| C16—C12—C13—C14 | -179.5 (2) | C13—C14—N4—N3 | 0.0 (2) |
| C12—C13—C14—N4 | -0.2 (3) | C15—C14—N4—N3 | -179.36 (18) |
| C12—C13—C14—C15 | 179.1 (2) | C12—N3—N4—C14 | 0.2 (2) |
| C2—C1—N1—C5 | -1.0 (3) | C11—N3—N4—C14 | -178.54 (18) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-------------------------------------|-------------|---------------|-----------------------|-------------------------|
| C10—H10 <i>B</i> ...O1 ⁱ | 0.96 | 2.56 | 3.435 (4) | 151 |

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