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5-(2-Methyl-5-nitrophenyl)-1H-tetrazole

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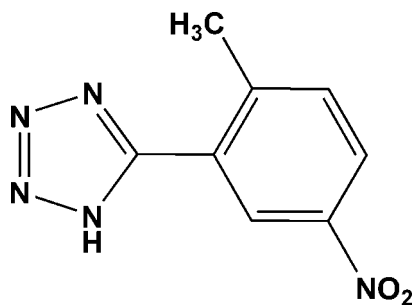
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.082; wR factor = 0.203; data-to-parameter ratio = 15.2.

In the title compound, $\text{C}_8\text{H}_7\text{N}_5\text{O}_2$, the benzene ring makes a dihedral angle of $45.7(2)^\circ$ with the tetrazole ring. In the crystal structure, the molecules are linked into a chain running along the a axis by $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, and the chains are linked through $\pi-\pi$ interactions between the tetrazole rings [centroid-centroid distance = $3.450(2)$ Å].

Related literature

For the use of tetrazole derivatives in coordination chemistry, see: Arp *et al.* (2000); Dai & Fu (2008); Wang *et al.* (2005); Xiong *et al.* (2002).



Experimental

Crystal data

$\text{C}_8\text{H}_7\text{N}_5\text{O}_2$
 $M_r = 205.19$

Monoclinic, $P2_1/c$
 $a = 4.9057(10)$ Å

$b = 16.938(3)$ Å
 $c = 11.463(2)$ Å
 $\beta = 98.65(3)^\circ$
 $V = 941.7(3)$ Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 298(2)$ K
 $0.25 \times 0.18 \times 0.15$ mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan
(*CrystalClear*, Rigaku, 2005)
 $T_{\min} = 0.971$, $T_{\max} = 0.977$

9281 measured reflections
2085 independent reflections
1434 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.082$
 $wR(F^2) = 0.203$
 $S = 1.13$
2085 reflections

137 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{N1}-\text{H1}\cdots\text{N4}^i$ | 0.86 | 1.98 | 2.775 (4) | 154 |

Symmetry code: (i) $x + 1, y, z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: CI2669).

References

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5-(2-Methyl-5-nitrophenyl)-1*H*-tetrazole

Jing Dai and Wei Dai

S1. Comment

Tetrazole derivatives have found wide range of applications in coordination chemistry because of their multiple coordination modes as ligands to metal ions and for the construction of novel metal-organic frameworks (Wang *et al.*, 2005; Xiong *et al.*, 2002). We report here the crystal structure of the title compound, 5-(2-methyl-5-nitrophenyl)-1*H*-tetrazole, (Fig.1).

The benzene ring makes a dihedral angle of 45.7 (2)° with the tetrazole ring owing to the C–C bond bridge which force the two rings to be twisted from each other. The bond distances and angles of the tetrazole rings are in the usual ranges (Wang *et al.*, 2005; Arp *et al.*, 2000; Dai & Fu, 2008).

The crystal packing is stabilized by N—H···N hydrogen bonds (Table 1), which link the molecules into chains running parallel to the *a* axis (Fig. 2). The adjacent chains are linked through π – π interactions between the tetrazole rings [centroid–centroid distance = 3.450 (2) Å] of the molecules at (*x*, *y*, *z*) and (1–*x*, 1–*y*, 1–*z*).

S2. Experimental

Under nitrogen protection, 2-methyl-5-nitrobenzotrile (30 mmol), NaN₃ (45 mmol) and NH₄Cl (33 mmol) were added in a flask and then DMF (50 ml) was added. The mixture was stirred at 383 K for 20 h, the resulting solution was poured into ice water (100 ml) and then hydrochloric acid (6 mol/l) was added to control the pH value to 6. The white solid obtained was filtered and washed with distilled water. The crude product was recrystallized with ethanol to obtain colourless block-shaped crystals of the title compound.

S3. Refinement

All H atoms were positioned geometrically and treated as riding with C—H = 0.93 Å (aromatic), 0.96 Å (methyl) and N—H = 0.86 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

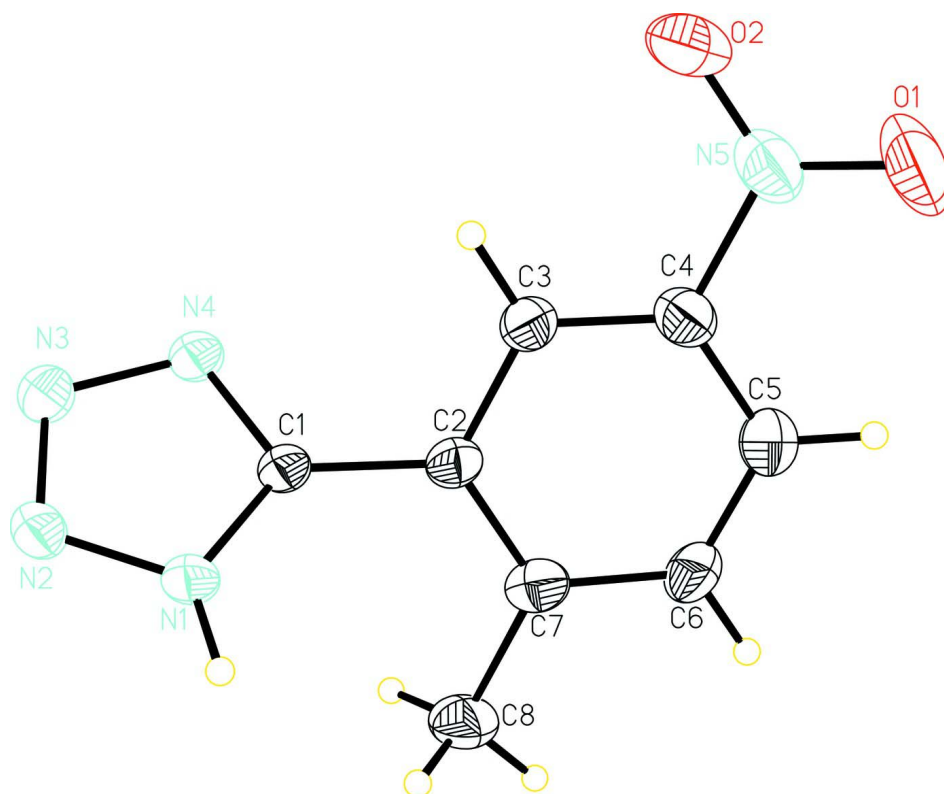


Figure 1

The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

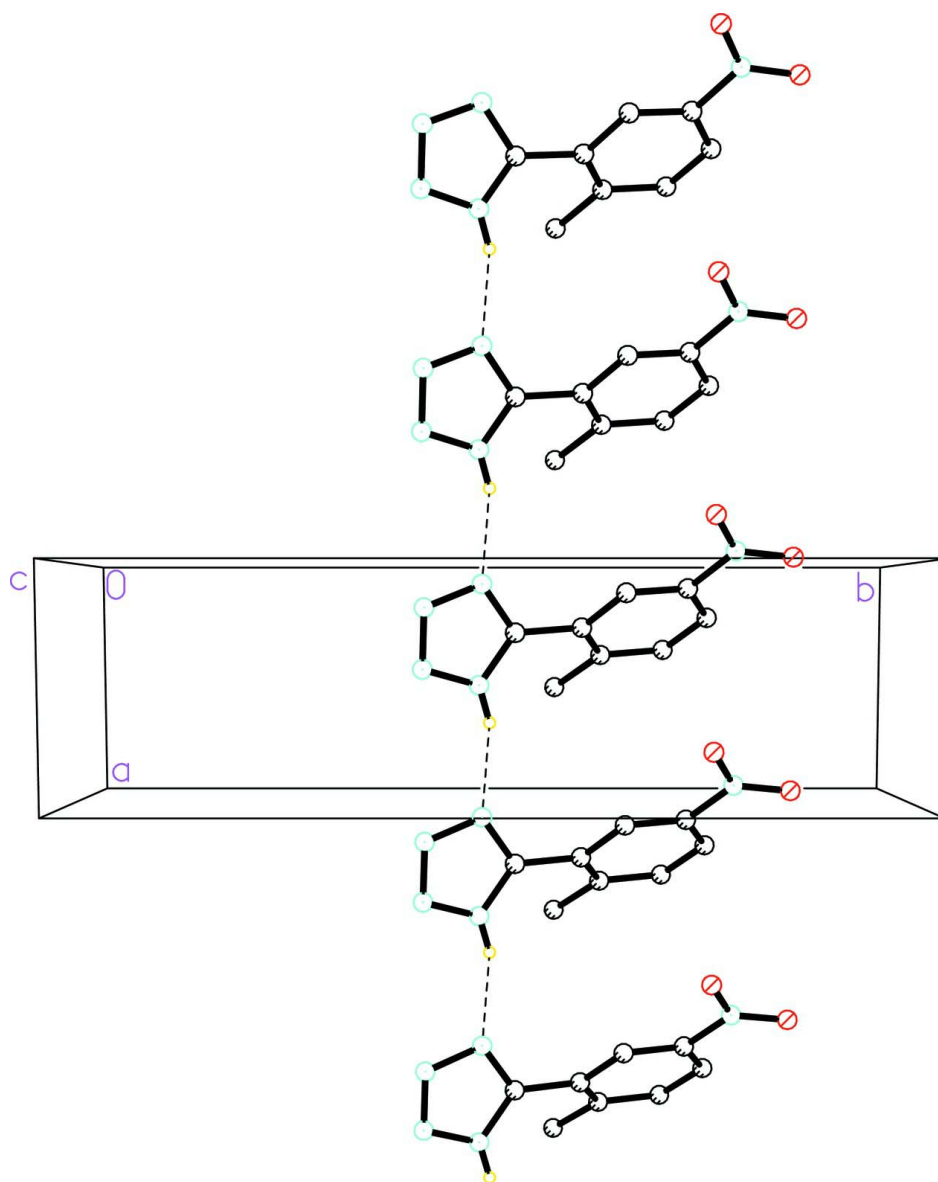


Figure 2

Part of the crystal packing of the title compound, showing a hydrogen-bonded (dashed lines) chain running along the *a* axis. H atoms not involved in hydrogen bonding have been omitted for clarity.

5-(2-Methyl-5-nitrophenyl)-1*H*-tetrazole

Crystal data

$C_8H_7N_5O_2$

$M_r = 205.19$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 4.9057(10)\ \text{\AA}$

$b = 16.938(3)\ \text{\AA}$

$c = 11.463(2)\ \text{\AA}$

$\beta = 98.65(3)^\circ$

$V = 941.7(3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 424$

$D_x = 1.447\ \text{Mg m}^{-3}$

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

Cell parameters from 1775 reflections

$\theta = 2.4\text{--}27.1^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 298$ K
Block, colourless

$0.25 \times 0.18 \times 0.15$ mm

Data collection

Rigaku Mercury2
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm⁻¹
 ω scans
Absorption correction: multi-scan
(*CrystalClear*, Rigaku, 2005)
 $T_{\min} = 0.971$, $T_{\max} = 0.977$

9281 measured reflections
2085 independent reflections
1434 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$
 $\theta_{\max} = 27.2^\circ$, $\theta_{\min} = 3.0^\circ$
 $h = -6 \rightarrow 6$
 $k = -21 \rightarrow 21$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.082$
 $wR(F^2) = 0.203$
 $S = 1.13$
2085 reflections
137 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
 $w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.9796P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ 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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|--------------|--------------|------------|----------------------------------|
| O1 | -0.0317 (10) | 0.86679 (19) | 0.3611 (3) | 0.1152 (15) |
| O2 | -0.2052 (8) | 0.7689 (2) | 0.4436 (3) | 0.0930 (12) |
| N1 | 0.5142 (5) | 0.48416 (16) | 0.3549 (2) | 0.0425 (7) |
| H1 | 0.6766 | 0.4970 | 0.3419 | 0.051* |
| N4 | 0.0825 (5) | 0.48791 (16) | 0.3638 (2) | 0.0416 (7) |
| N3 | 0.1836 (6) | 0.41763 (16) | 0.4075 (3) | 0.0473 (7) |
| C1 | 0.2904 (6) | 0.52855 (17) | 0.3320 (3) | 0.0360 (7) |
| N2 | 0.4451 (6) | 0.41548 (16) | 0.4018 (3) | 0.0470 (7) |
| C2 | 0.2681 (6) | 0.61097 (19) | 0.2897 (3) | 0.0397 (7) |
| C7 | 0.3890 (7) | 0.6358 (2) | 0.1932 (3) | 0.0485 (9) |
| C3 | 0.1193 (7) | 0.6641 (2) | 0.3495 (3) | 0.0426 (8) |
| H3 | 0.0349 | 0.6473 | 0.4125 | 0.051* |
| C4 | 0.1007 (7) | 0.7403 (2) | 0.3140 (3) | 0.0506 (9) |
| C6 | 0.3672 (8) | 0.7152 (2) | 0.1624 (3) | 0.0565 (10) |

| | | | | |
|-----|-------------|------------|------------|-------------|
| H6 | 0.4510 | 0.7333 | 0.0999 | 0.068* |
| N5 | -0.0555 (8) | 0.7961 (2) | 0.3775 (3) | 0.0676 (10) |
| C5 | 0.2254 (9) | 0.7676 (2) | 0.2220 (4) | 0.0628 (11) |
| H5 | 0.2136 | 0.8206 | 0.2006 | 0.075* |
| C8 | 0.5334 (9) | 0.5794 (2) | 0.1215 (4) | 0.0628 (11) |
| H8A | 0.4164 | 0.5347 | 0.0992 | 0.094* |
| H8B | 0.7020 | 0.5618 | 0.1677 | 0.094* |
| H8C | 0.5738 | 0.6056 | 0.0519 | 0.094* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|--------------|-------------|-------------|
| O1 | 0.197 (4) | 0.0494 (19) | 0.105 (3) | 0.033 (2) | 0.042 (3) | 0.0013 (19) |
| O2 | 0.107 (3) | 0.089 (2) | 0.095 (3) | 0.032 (2) | 0.054 (2) | 0.003 (2) |
| N1 | 0.0275 (13) | 0.0435 (15) | 0.0576 (18) | 0.0007 (12) | 0.0105 (12) | 0.0030 (13) |
| N4 | 0.0317 (13) | 0.0391 (14) | 0.0558 (17) | -0.0045 (11) | 0.0129 (12) | 0.0006 (13) |
| N3 | 0.0422 (16) | 0.0397 (15) | 0.0616 (19) | -0.0060 (13) | 0.0132 (14) | 0.0008 (14) |
| C1 | 0.0293 (15) | 0.0369 (16) | 0.0428 (17) | -0.0034 (13) | 0.0090 (13) | 0.0002 (14) |
| N2 | 0.0434 (16) | 0.0359 (14) | 0.0637 (19) | 0.0033 (12) | 0.0144 (14) | 0.0005 (13) |
| C2 | 0.0305 (15) | 0.0437 (17) | 0.0455 (18) | -0.0033 (13) | 0.0076 (13) | 0.0006 (15) |
| C7 | 0.0400 (18) | 0.061 (2) | 0.0447 (19) | 0.0012 (16) | 0.0085 (15) | 0.0040 (17) |
| C3 | 0.0394 (17) | 0.0451 (18) | 0.0443 (19) | 0.0003 (14) | 0.0096 (14) | 0.0048 (15) |
| C4 | 0.051 (2) | 0.049 (2) | 0.052 (2) | 0.0093 (16) | 0.0103 (17) | 0.0000 (17) |
| C6 | 0.061 (2) | 0.055 (2) | 0.057 (2) | 0.0009 (19) | 0.0216 (19) | 0.0211 (19) |
| N5 | 0.086 (3) | 0.055 (2) | 0.062 (2) | 0.0235 (19) | 0.013 (2) | 0.0028 (18) |
| C5 | 0.070 (3) | 0.050 (2) | 0.071 (3) | 0.008 (2) | 0.019 (2) | 0.016 (2) |
| C8 | 0.067 (3) | 0.072 (3) | 0.056 (2) | 0.012 (2) | 0.029 (2) | 0.000 (2) |

Geometric parameters (Å, °)

| | | | |
|----------|-----------|----------|-----------|
| O1—N5 | 1.220 (5) | C7—C8 | 1.505 (5) |
| O2—N5 | 1.222 (5) | C3—C4 | 1.352 (5) |
| N1—C1 | 1.324 (4) | C3—H3 | 0.93 |
| N1—N2 | 1.346 (4) | C4—C5 | 1.377 (5) |
| N1—H1 | 0.86 | C4—N5 | 1.477 (5) |
| N4—C1 | 1.326 (4) | C6—C5 | 1.371 (5) |
| N4—N3 | 1.357 (4) | C6—H6 | 0.93 |
| N3—N2 | 1.294 (4) | C5—H5 | 0.93 |
| C1—C2 | 1.477 (4) | C8—H8A | 0.96 |
| C2—C7 | 1.396 (5) | C8—H8B | 0.96 |
| C2—C3 | 1.401 (4) | C8—H8C | 0.96 |
| C7—C6 | 1.391 (5) | | |
| C1—N1—N2 | 108.6 (2) | C3—C4—C5 | 122.2 (3) |
| C1—N1—H1 | 125.7 | C3—C4—N5 | 118.7 (3) |
| N2—N1—H1 | 125.7 | C5—C4—N5 | 119.1 (3) |
| C1—N4—N3 | 107.7 (2) | C5—C6—C7 | 121.7 (3) |
| N2—N3—N4 | 108.4 (2) | C5—C6—H6 | 119.2 |

| | | | |
|-------------|------------|-------------|------------|
| N1—C1—N4 | 107.5 (3) | C7—C6—H6 | 119.2 |
| N1—C1—C2 | 128.3 (3) | O1—N5—O2 | 123.2 (4) |
| N4—C1—C2 | 124.0 (3) | O1—N5—C4 | 118.9 (4) |
| N3—N2—N1 | 107.9 (3) | O2—N5—C4 | 117.9 (3) |
| C7—C2—C3 | 120.6 (3) | C6—C5—C4 | 118.8 (4) |
| C7—C2—C1 | 121.7 (3) | C6—C5—H5 | 120.6 |
| C3—C2—C1 | 117.7 (3) | C4—C5—H5 | 120.6 |
| C6—C7—C2 | 117.8 (3) | C7—C8—H8A | 109.5 |
| C6—C7—C8 | 120.0 (3) | C7—C8—H8B | 109.5 |
| C2—C7—C8 | 122.2 (3) | H8A—C8—H8B | 109.5 |
| C4—C3—C2 | 118.9 (3) | C7—C8—H8C | 109.5 |
| C4—C3—H3 | 120.6 | H8A—C8—H8C | 109.5 |
| C2—C3—H3 | 120.6 | H8B—C8—H8C | 109.5 |
| | | | |
| C1—N4—N3—N2 | -0.4 (4) | C1—C2—C7—C8 | 4.1 (5) |
| N2—N1—C1—N4 | -0.3 (4) | C7—C2—C3—C4 | -1.6 (5) |
| N2—N1—C1—C2 | 174.4 (3) | C1—C2—C3—C4 | 178.6 (3) |
| N3—N4—C1—N1 | 0.4 (4) | C2—C3—C4—C5 | -0.7 (6) |
| N3—N4—C1—C2 | -174.5 (3) | C2—C3—C4—N5 | -179.8 (3) |
| N4—N3—N2—N1 | 0.2 (4) | C2—C7—C6—C5 | -1.9 (6) |
| C1—N1—N2—N3 | 0.1 (4) | C8—C7—C6—C5 | 176.7 (4) |
| N1—C1—C2—C7 | 49.1 (5) | C3—C4—N5—O1 | 166.6 (4) |
| N4—C1—C2—C7 | -137.0 (3) | C5—C4—N5—O1 | -12.5 (6) |
| N1—C1—C2—C3 | -131.2 (3) | C3—C4—N5—O2 | -14.0 (6) |
| N4—C1—C2—C3 | 42.7 (5) | C5—C4—N5—O2 | 166.9 (4) |
| C3—C2—C7—C6 | 2.9 (5) | C7—C6—C5—C4 | -0.4 (6) |
| C1—C2—C7—C6 | -177.4 (3) | C3—C4—C5—C6 | 1.7 (6) |
| C3—C2—C7—C8 | -175.7 (3) | N5—C4—C5—C6 | -179.2 (4) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|--------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N1—H1 \cdots N4 ⁱ | 0.86 | 1.98 | 2.775 (4) | 154 |

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