

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-(4-Nitrobenzyl)pyridine

Deeb Taher, Firas F. Awwadi and Mohammed H. Kailani*

Department of Chemistry, The University of Jordan, Amman 11942, Jordan

Correspondence e-mail: kailani@ju.edu.jo

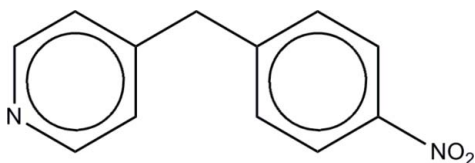
Received 27 May 2013; accepted 20 June 2013

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.047; wR factor = 0.106; data-to-parameter ratio = 14.7.

The title compound, $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_2$, has a twisted conformation, with a dihedral angle between the planes of the pyridine and benzene rings of 78.4 (2)°. The nitro group is coplanar with the attached benzene ring within experimental error. The molecules form centrosymmetric dimers *via* $\text{C}_{\text{ar}}-\text{H}\cdots\text{O}$ interactions ($\text{H}\cdots\text{O} = 2.49$ Å) and the dimers are π -stacked along the b axis [the separation between ring centroids is 3.788 (2) Å].

Related literature

For adducts of the title compound with different organic acids, see: Smith *et al.* (1997); Smith & Wermuth (2010, 2013). For a zinc complex of the title compound, see: Smith *et al.* (2011). For the analysis of π -stacking interactions, see: Dolomanov *et al.* (2009).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_2$
 $M_r = 214.22$
 Monoclinic, $P2_1/c$

$a = 11.4138$ (9) Å
 $b = 6.1241$ (5) Å
 $c = 15.5812$ (13) Å

$\beta = 104.561$ (9)°
 $V = 1054.13$ (15) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.4 \times 0.2 \times 0.15$ mm

Data collection

Agilent Xcalibur Eos diffractometer
 Absorption correction: multi-scan
 (*CrysAlis PRO*; Agilent, 2011)
 $T_{\text{min}} = 0.770$, $T_{\text{max}} = 1.000$

4351 measured reflections
 2136 independent reflections
 1514 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.106$
 $S = 1.03$
 2136 reflections

145 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.12$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C9}-\text{H9A}\cdots\text{O2}^i$	0.93	2.49	3.302 (2)	146

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

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: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

The authors thank the University of Jordan and Hamdi Mango Center for Scientific Research for providing support and time to collect the single-crystal X-ray diffraction data set.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LD2106).

References

- Agilent (2011). *CrysAlis PRO*. Agilent Technologies, Yarnton, Oxfordshire, England.
 Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Smith, G., Lynch, D. E., Byriel, K. A. & Kennard, C. H. L. (1997). *J. Chem. Crystallogr.* **27**, 307–317.
 Smith, G. & Wermuth, U. D. (2010). *Acta Cryst.* **E66**, o1173.
 Smith, G. & Wermuth, U. D. (2013). *Acta Cryst.* **E69**, o206.
 Smith, G., Wermuth, U. D. & Williams, M. L. (2011). *Acta Cryst.* **E67**, m359.

supporting information

Acta Cryst. (2013). E69, o1164 [https://doi.org/10.1107/S1600536813017145]

4-(4-Nitrobenzyl)pyridine

Deeb Taher, Firas F. Awwadi and Mohammed H. Kailani

S1. Comment

X-Ray structure of the title compound was never reported before in its non-coordinated form, even though several works have been published on its pyridinium salts/adducts. The adducts with carboxylic acids were reported for 4-amino-benzoic (Smith *et al.*, 1997) and 5-nitrosalicylic acid (Smith & Wermuth, 2010). Recently, a structure of an adduct with 3-carboxy-4-hydroxybenzenesulfonic acid was also determined (Smith & Wermuth, 2013). The structures of the adducts are dominated by *N*(pyridine)—H \cdots O hydrogen bonding interactions. In addition, X-ray structure of a zinc complex of the title compound (Diiodidobis[4-(4-nitrobenzyl)pyridine- κ N¹]zinc) has also been determined (Smith *et al.*, 2011).

The title compound (Fig. 1) gives colorless crystals. The angle between the planes of benzene and pyridine rings is 78.43° and the nitro group is coplanar with the benzene ring. The two aromatic planes are twisted relative to each other, which result in reduction of molecular symmetry from C_s to C₁: the dihedral angle C2—C3 \cdots C7—C8 is 30.5 (2)°. Two molecular units of the title compound inter-associate through duplex C9—H \cdots O2 hydrogen bonds to form a cyclic dimer (Fig. 2 and Table 1). Then, these dimers are stacked *via* $\pi\cdots\pi$ interactions between benzene rings to form ribbon structure extending parallel to *b*-axis (Fig. 3); the angle between the two planes, centroid-centroid distance and shift distance are 0°, 3.788 Å and 1.613 Å, respectively, as determined by Olex2 program package (Dolomanov *et al.*, 2009). Subsequently, these ribbons are interdigitated to form the final three-dimensional structure (Fig. 4).

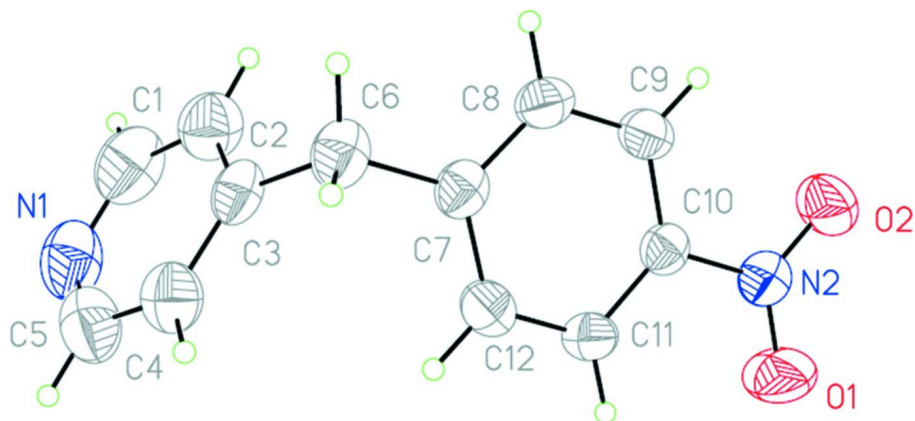
The nitro group of the title compound, was found to be a major factor in determining the interactions in the crystal form, unlike in the previously published structures where the pyridinic nitrogen was the main driving force for amolecular association.

S2. Experimental

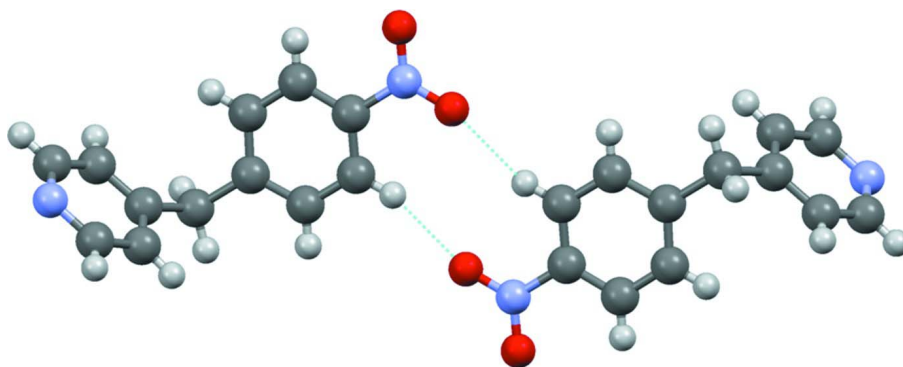
Crystals of the title compound were obtained by dissolving 1 mmol of 4-(4-nitrobenzyl) pyridine in 30 ml of hot 96% ethanol. Partial evaporation of the hot-filtered solution at room temperature yielded colourless crystals from which a block section was cleaved for the X-ray analysis.

S3. Refinement

The structure was solved by direct methods and refined by least squares method on F₂ using the *SHELXTL* program package. All atoms were refined anisotropically. Hydrogen atoms were placed at the calculated positions using a riding model with C(aromatic)—H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, and with C(aliphatic)—H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. refinement details)

**Figure 1**

Molecular unit of the title compound. Thermal ellipsoids are shown at 50% probability.

**Figure 2**

Structure of the cyclic dimer.

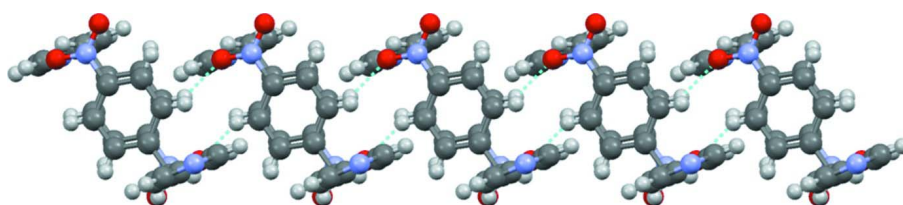
**Figure 3**

Illustration of the ribbon structure of the title compound.

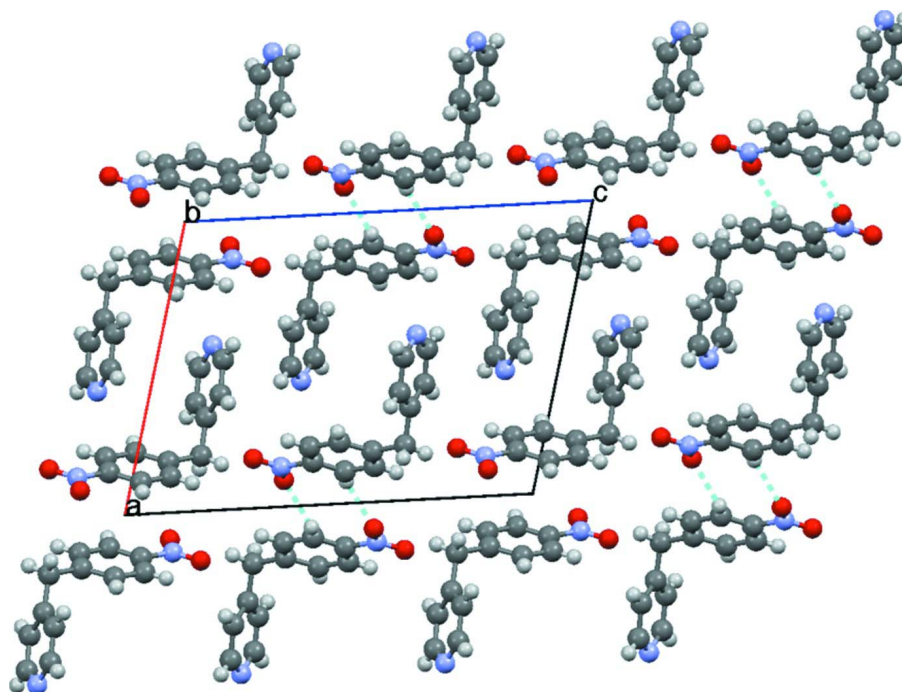


Figure 4

Illustration of the three dimensional structure of the title compound viewed along the *b*-axis.

4-(4-Nitrobenzyl)pyridine

Crystal data

$C_{12}H_{10}N_2O_2$

$M_r = 214.22$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.4138 (9) \text{ \AA}$

$b = 6.1241 (5) \text{ \AA}$

$c = 15.5812 (13) \text{ \AA}$

$\beta = 104.561 (9)^\circ$

$V = 1054.13 (15) \text{ \AA}^3$

$Z = 4$

$F(000) = 448$

$D_x = 1.350 \text{ Mg m}^{-3}$

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

Cell parameters from 949 reflections

$\theta = 3.3\text{--}29.0^\circ$

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

$T = 293 \text{ K}$

Needle, white

$0.4 \times 0.2 \times 0.15 \text{ mm}$

Data collection

Agilent Xcalibur Eos
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: $16.0534 \text{ pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Agilent, 2011)

$T_{\min} = 0.770$, $T_{\max} = 1.000$

4351 measured reflections

2136 independent reflections

1514 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 26.3^\circ$, $\theta_{\min} = 3.6^\circ$

$h = -13 \rightarrow 14$

$k = -7 \rightarrow 6$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.106$
 $S = 1.03$
 2136 reflections
 145 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
 $w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.1216P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.12 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. Absorption correction CrysAlis PRO (Agilent, 2011). Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N2	0.13729 (12)	0.2522 (2)	0.13717 (9)	0.0459 (4)
C10	0.15049 (13)	0.1215 (2)	0.06150 (10)	0.0361 (4)
C9	0.11079 (14)	0.2054 (3)	-0.02263 (11)	0.0413 (4)
H9A	0.0757	0.3433	-0.0317	0.050*
C7	0.17562 (14)	-0.1248 (3)	-0.08069 (10)	0.0391 (4)
O2	0.09206 (12)	0.4333 (2)	0.12370 (9)	0.0633 (4)
C11	0.20080 (14)	-0.0837 (3)	0.07671 (11)	0.0410 (4)
H11A	0.2258	-0.1390	0.1340	0.049*
C8	0.12404 (14)	0.0808 (3)	-0.09329 (11)	0.0427 (4)
H8A	0.0978	0.1361	-0.1505	0.051*
C12	0.21313 (14)	-0.2047 (3)	0.00541 (11)	0.0426 (4)
H12A	0.2473	-0.3433	0.0149	0.051*
C6	0.19074 (15)	-0.2601 (3)	-0.15842 (11)	0.0496 (5)
H6A	0.1457	-0.3953	-0.1607	0.059*
H6B	0.1576	-0.1809	-0.2130	0.059*
O1	0.17074 (16)	0.1747 (2)	0.21073 (9)	0.0835 (5)
C3	0.32183 (15)	-0.3119 (3)	-0.15136 (10)	0.0445 (4)
C4	0.37349 (17)	-0.5080 (3)	-0.11888 (12)	0.0555 (5)
H4A	0.3263	-0.6168	-0.1028	0.067*
N1	0.56897 (15)	-0.3995 (3)	-0.13144 (12)	0.0712 (5)
C2	0.39748 (17)	-0.1617 (3)	-0.17558 (13)	0.0610 (5)
H2A	0.3676	-0.0274	-0.1992	0.073*
C5	0.49444 (19)	-0.5428 (4)	-0.11034 (14)	0.0670 (6)

H5A	0.5263	-0.6770	-0.0881	0.080*
C1	0.51831 (19)	-0.2128 (4)	-0.16440 (15)	0.0731 (6)
H1B	0.5676	-0.1088	-0.1812	0.088*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0485 (9)	0.0470 (9)	0.0436 (9)	0.0015 (7)	0.0142 (7)	-0.0001 (7)
C10	0.0327 (8)	0.0392 (9)	0.0376 (9)	-0.0024 (7)	0.0108 (7)	0.0001 (7)
C9	0.0387 (9)	0.0401 (9)	0.0448 (10)	0.0031 (7)	0.0098 (8)	0.0074 (8)
C7	0.0308 (8)	0.0477 (10)	0.0398 (9)	-0.0043 (7)	0.0104 (7)	-0.0016 (8)
O2	0.0808 (10)	0.0477 (8)	0.0618 (9)	0.0179 (7)	0.0189 (7)	-0.0018 (7)
C11	0.0430 (9)	0.0428 (9)	0.0363 (9)	0.0032 (8)	0.0086 (8)	0.0070 (8)
C8	0.0424 (9)	0.0498 (10)	0.0350 (9)	-0.0003 (8)	0.0082 (8)	0.0064 (8)
C12	0.0396 (9)	0.0385 (9)	0.0494 (10)	0.0037 (7)	0.0109 (8)	0.0026 (8)
C6	0.0413 (10)	0.0621 (11)	0.0455 (10)	-0.0024 (8)	0.0113 (8)	-0.0088 (9)
O1	0.1380 (14)	0.0749 (10)	0.0378 (8)	0.0326 (10)	0.0224 (8)	0.0078 (7)
C3	0.0436 (9)	0.0551 (11)	0.0361 (9)	-0.0042 (9)	0.0126 (8)	-0.0120 (8)
C4	0.0519 (11)	0.0561 (11)	0.0601 (12)	-0.0023 (9)	0.0172 (10)	-0.0061 (10)
N1	0.0481 (10)	0.0950 (14)	0.0719 (12)	0.0042 (10)	0.0176 (9)	-0.0145 (11)
C2	0.0540 (12)	0.0668 (13)	0.0653 (13)	-0.0035 (10)	0.0211 (10)	0.0019 (11)
C5	0.0585 (13)	0.0708 (14)	0.0692 (14)	0.0117 (12)	0.0114 (11)	-0.0119 (11)
C1	0.0535 (13)	0.0956 (17)	0.0759 (15)	-0.0169 (13)	0.0271 (12)	-0.0053 (14)

Geometric parameters (Å, °)

N2—O1	1.2101 (17)	C6—C3	1.506 (2)
N2—O2	1.2194 (17)	C6—H6A	0.9700
N2—C10	1.464 (2)	C6—H6B	0.9700
C10—C9	1.374 (2)	C3—C4	1.377 (2)
C10—C11	1.377 (2)	C3—C2	1.378 (2)
C9—C8	1.379 (2)	C4—C5	1.370 (3)
C9—H9A	0.9300	C4—H4A	0.9300
C7—C8	1.383 (2)	N1—C5	1.320 (3)
C7—C12	1.391 (2)	N1—C1	1.325 (3)
C7—C6	1.513 (2)	C2—C1	1.382 (3)
C11—C12	1.372 (2)	C2—H2A	0.9300
C11—H11A	0.9300	C5—H5A	0.9300
C8—H8A	0.9300	C1—H1B	0.9300
C12—H12A	0.9300		
O1—N2—O2	122.59 (15)	C3—C6—H6A	109.3
O1—N2—C10	118.47 (14)	C7—C6—H6A	109.3
O2—N2—C10	118.93 (14)	C3—C6—H6B	109.3
C9—C10—C11	121.90 (15)	C7—C6—H6B	109.3
C9—C10—N2	119.20 (14)	H6A—C6—H6B	108.0
C11—C10—N2	118.89 (14)	C4—C3—C2	116.31 (17)
C10—C9—C8	118.60 (15)	C4—C3—C6	122.41 (16)

C10—C9—H9A	120.7	C2—C3—C6	121.27 (17)
C8—C9—H9A	120.7	C5—C4—C3	119.94 (18)
C8—C7—C12	118.32 (15)	C5—C4—H4A	120.0
C8—C7—C6	121.02 (15)	C3—C4—H4A	120.0
C12—C7—C6	120.66 (15)	C5—N1—C1	115.11 (18)
C12—C11—C10	118.48 (15)	C3—C2—C1	119.2 (2)
C12—C11—H11A	120.8	C3—C2—H2A	120.4
C10—C11—H11A	120.8	C1—C2—H2A	120.4
C9—C8—C7	121.25 (15)	N1—C5—C4	124.7 (2)
C9—C8—H8A	119.4	N1—C5—H5A	117.6
C7—C8—H8A	119.4	C4—C5—H5A	117.6
C11—C12—C7	121.44 (15)	N1—C1—C2	124.7 (2)
C11—C12—H12A	119.3	N1—C1—H1B	117.7
C7—C12—H12A	119.3	C2—C1—H1B	117.7
C3—C6—C7	111.59 (13)		
O1—N2—C10—C9	178.75 (15)	C6—C7—C12—C11	179.59 (14)
O2—N2—C10—C9	-0.5 (2)	C8—C7—C6—C3	119.47 (17)
O1—N2—C10—C11	-0.3 (2)	C12—C7—C6—C3	-60.7 (2)
O2—N2—C10—C11	-179.55 (15)	C7—C6—C3—C4	98.12 (19)
C11—C10—C9—C8	-1.2 (2)	C7—C6—C3—C2	-80.4 (2)
N2—C10—C9—C8	179.84 (13)	C2—C3—C4—C5	1.5 (3)
C9—C10—C11—C12	1.2 (2)	C6—C3—C4—C5	-177.18 (16)
N2—C10—C11—C12	-179.81 (13)	C4—C3—C2—C1	-1.4 (3)
C10—C9—C8—C7	0.2 (2)	C6—C3—C2—C1	177.24 (17)
C12—C7—C8—C9	0.6 (2)	C1—N1—C5—C4	-1.2 (3)
C6—C7—C8—C9	-179.57 (14)	C3—C4—C5—N1	-0.1 (3)
C10—C11—C12—C7	-0.3 (2)	C5—N1—C1—C2	1.3 (3)
C8—C7—C12—C11	-0.6 (2)	C3—C2—C1—N1	0.0 (3)

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>
C9—H9A \cdots O2 ⁱ	0.93	2.49	3.302 (2)	146

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