Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(4-Methylpiperazin-1-yl)isobenzofuran-1(3*H*)-one[1]

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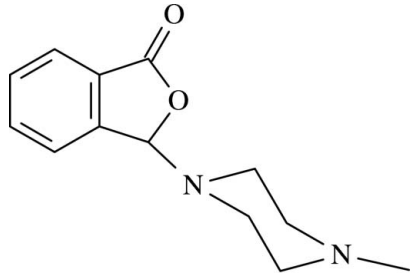
Received 26 March 2008; accepted 26 March 2008

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.091; data-to-parameter ratio = 15.4.

In the molecule of the title compound, $C_{13}H_{16}N_2O_2$, the phthalide ring system is virtually planar, with a dihedral angle between the fused five- and six-membered rings of 1.17 (4)°. The methylpiperazine ring adopts a chair conformation. In the crystal structure, intermolecular C—H···O and C—H···N hydrogen bonds link the molecules, generating edge-fused $R_3^3(17)$ ring motifs, to form a three-dimensional network.

Related literature

For a related structure, see: Odabaşoğlu & Büyükgüngör (2006). For ring motif details, see: Bernstein *et al.* (1995); Etter (1990). For ring conformation puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{13}H_{16}N_2O_2$ M_r = 232.28
Monoclinic, $P2_1/c$
a = 13.1442 (7) Å
b = 6.0567 (4) Å
c = 15.7845 (10) Å
β = 104.022 (5)°
V = 1219.17 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.56 × 0.49 × 0.37 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.952, T_{max} = 0.969
14223 measured reflections
2394 independent reflections
1890 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.091
S = 1.04
2394 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1^{i}	0.98	2.69	3.6135 (18)	157
C10—H10A···N2^{ii}	0.97	2.60	3.5315 (17)	160

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDSII diffractometer (purchased under grant F.279 of the University Research Fund).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2441).

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[1] 3-Substituted phthalides. XXXVI.

supporting information

Acta Cryst. (2008). E**64**, o779 [doi:10.1107/S1600536808008209]

3-(4-Methylpiperazin-1-yl)isobenzofuran-1(3*H*)-one

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S1. Comment

The present work is part of a structural study of compounds of 3-substituted phthalides, and we report herein the structure of the title compound, (I).

In the molecule of (I), (Fig. 1), rings A (C2–C7) and B (C1/C2/C7/C8/O2) are, of course, planar. The dihedral angle between them is A/B = 1.17 (4)°. So, rings A and B are also nearly coplanar. Ring C (N1/N2/C9–C12) is not planar, having total puckering amplitude, Q_T, of 1.014 (3) Å. It adopts chair [φ = 29.44 (2)° and θ = 59.51 (3)°] conformation (Cremer & Pople, 1975).

In the crystal structure, intermolecular C—H···O and C—H···N hydrogen bonds (Table 1) link the molecules, generating edge-fused $R_3^3(17)$ (Fig. 2) ring motifs (Bernstein *et al.*, 1995; Etter, 1990), to form a three-dimensional network, in which they may be effective in the stabilization of the structure.

S2. Experimental

The title compound was prepared according to the method described by Odabaşoğlu & Büyükgüngör (2006), using phthalaldehydic acid and 1-methylpiperazine as starting materials (yield; 85%). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol–DMF (1:1) solution at room temperature.

S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93, 0.98, 0.97 and 0.96 Å for aromatic, methine, methylene and methyl H, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = xU_{eq}(C)$, where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

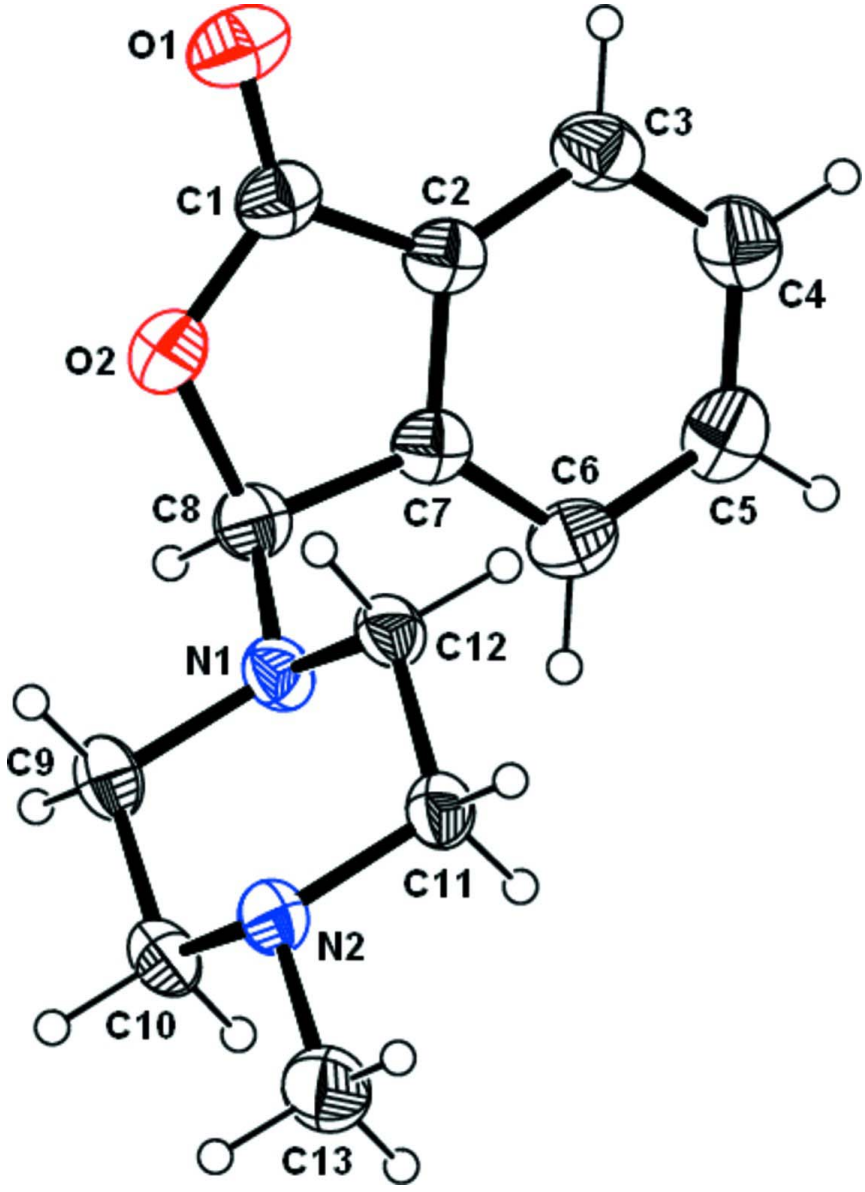

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

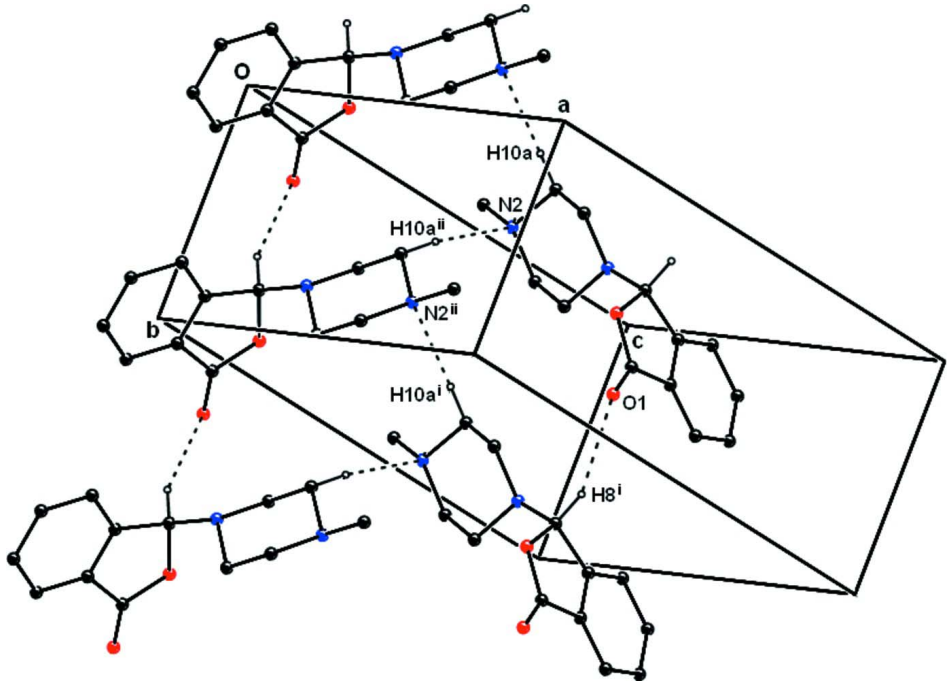

Figure 2

A partial packing diagram of (I), showing the formation of $R_3^3(17)$ ring motifs. Hydrogen bonds are shown as dashed lines [symmetry codes: (i) *x*, *y* + 1, *z*; (ii) 1 - *x*, 2 - *y*, 1/2 - *z*]. H atoms not involved in hydrogen bondings have been omitted for clarity.

3-(4-methylpiperazin-1-yl)isobenzofuran-1(3*H*)-one

Crystal data

$C_{13}H_{16}N_2O_2$
$M_r = 232.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.1442\ (7)$ Å
$b = 6.0567\ (4)$ Å
$c = 15.7845\ (10)$ Å
$\beta = 104.022\ (5)°$
$V = 1219.17\ (13)$ Å^3
$Z = 4$

$F(000) = 496$
$D_x = 1.265$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 14223 reflections
$\theta = 1.3–27.2°$
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Prism, colourless
0.56 × 0.49 × 0.37 mm

Data collection

Stoe IPDSII diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
w–scan rotation method
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)

$T_{min} = 0.952$, $T_{max} = 0.969$
14223 measured reflections
2394 independent reflections
1890 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.6°$
$h = -16\rightarrow16$
$k = -7\rightarrow7$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.091$
$S = 1.04$
2394 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.1087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.052 (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 $F^2 > 2\text{sigma}(F^2)$ 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	0.90768 (9)	0.78386 (19)	0.39052 (8)	0.0726 (3)
O2	0.83029 (7)	0.45298 (17)	0.38597 (6)	0.0573 (3)
N1	0.69300 (8)	0.23856 (17)	0.42651 (7)	0.0465 (3)
N2	0.47776 (8)	0.18458 (18)	0.34023 (6)	0.0468 (3)
C1	0.87740 (10)	0.6335 (2)	0.42833 (9)	0.0536 (3)
C2	0.88098 (9)	0.6114 (2)	0.52149 (9)	0.0500 (3)
C3	0.91995 (11)	0.7553 (3)	0.59001 (11)	0.0616 (4)
H3	0.9512	0.8881	0.5810	0.074*
C4	0.91066 (11)	0.6944 (3)	0.67175 (11)	0.0678 (4)
H4	0.9355	0.7880	0.7189	0.081*
C5	0.86475 (11)	0.4955 (3)	0.68471 (10)	0.0656 (4)
H5	0.8597	0.4574	0.7406	0.079*
C6	0.82633 (10)	0.3527 (3)	0.61639 (9)	0.0582 (4)
H6	0.7954	0.2194	0.6254	0.070*
C7	0.83528 (9)	0.4141 (2)	0.53414 (9)	0.0479 (3)
C8	0.80023 (10)	0.2952 (2)	0.44870 (9)	0.0502 (3)
H8	0.8419	0.1601	0.4509	0.060*
C9	0.66011 (11)	0.0825 (2)	0.35430 (9)	0.0520 (3)
H9A	0.7084	−0.0412	0.3621	0.062*
H9B	0.6608	0.1545	0.2995	0.062*
C10	0.55130 (11)	0.0013 (2)	0.35199 (9)	0.0515 (3)
H10A	0.5293	−0.1029	0.3044	0.062*
H10B	0.5515	−0.0746	0.4061	0.062*

C11	0.51096 (10)	0.3423 (2)	0.41085 (9)	0.0485 (3)
H11A	0.5086	0.2729	0.4657	0.058*
H11B	0.4629	0.4664	0.4017	0.058*
C12	0.62040 (10)	0.4247 (2)	0.41613 (9)	0.0468 (3)
H12A	0.6219	0.5054	0.3634	0.056*
H12B	0.6417	0.5244	0.4653	0.056*
C13	0.37178 (12)	0.1092 (3)	0.33673 (11)	0.0711 (5)
H13A	0.3252	0.2336	0.3289	0.107*
H13B	0.3706	0.0351	0.3903	0.107*
H13C	0.3496	0.0089	0.2887	0.107*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0645 (7)	0.0696 (7)	0.0885 (8)	0.0008 (5)	0.0281 (6)	0.0230 (6)
O2	0.0522 (5)	0.0658 (6)	0.0575 (6)	0.0024 (5)	0.0200 (4)	0.0016 (5)
N1	0.0447 (6)	0.0395 (6)	0.0549 (6)	0.0040 (5)	0.0116 (5)	−0.0060 (5)
N2	0.0493 (6)	0.0476 (6)	0.0426 (6)	−0.0004 (5)	0.0095 (4)	−0.0034 (5)
C1	0.0395 (7)	0.0542 (8)	0.0687 (9)	0.0076 (6)	0.0164 (6)	0.0087 (7)
C2	0.0346 (6)	0.0504 (8)	0.0633 (8)	0.0055 (6)	0.0086 (6)	0.0033 (6)
C3	0.0409 (7)	0.0587 (9)	0.0816 (10)	−0.0033 (6)	0.0082 (7)	−0.0051 (8)
C4	0.0453 (8)	0.0845 (12)	0.0679 (10)	−0.0015 (8)	0.0024 (7)	−0.0171 (9)
C5	0.0471 (8)	0.0933 (12)	0.0535 (8)	0.0038 (8)	0.0066 (6)	0.0013 (8)
C6	0.0472 (8)	0.0642 (9)	0.0619 (8)	−0.0007 (7)	0.0106 (6)	0.0092 (7)
C7	0.0372 (6)	0.0490 (8)	0.0558 (8)	0.0042 (6)	0.0080 (5)	0.0032 (6)
C8	0.0479 (7)	0.0448 (7)	0.0588 (8)	0.0064 (6)	0.0148 (6)	0.0027 (6)
C9	0.0587 (8)	0.0429 (7)	0.0544 (8)	0.0107 (6)	0.0140 (6)	−0.0077 (6)
C10	0.0651 (8)	0.0407 (7)	0.0463 (7)	−0.0012 (6)	0.0087 (6)	−0.0069 (6)
C11	0.0495 (7)	0.0466 (7)	0.0514 (7)	0.0025 (6)	0.0165 (6)	−0.0073 (6)
C12	0.0477 (7)	0.0372 (7)	0.0573 (8)	0.0032 (6)	0.0159 (6)	−0.0065 (6)
C13	0.0568 (9)	0.0815 (11)	0.0733 (10)	−0.0138 (8)	0.0123 (7)	−0.0175 (9)

Geometric parameters (Å, º)

C1—O1	1.2078 (17)	C9—C10	1.504 (2)
C1—O2	1.3512 (17)	C9—H9A	0.9700
C1—C2	1.466 (2)	C9—H9B	0.9700
C2—C7	1.3740 (19)	C10—N2	1.4537 (17)
C2—C3	1.387 (2)	C10—H10A	0.9700
C3—C4	1.375 (2)	C10—H10B	0.9700
C3—H3	0.9300	C11—N2	1.4526 (16)
C4—C5	1.384 (2)	C11—C12	1.5056 (18)
C4—H4	0.9300	C11—H11A	0.9700
C5—C6	1.379 (2)	C11—H11B	0.9700
C5—H5	0.9300	C12—N1	1.4602 (16)
C6—C7	1.3826 (19)	C12—H12A	0.9700
C6—H6	0.9300	C12—H12B	0.9700
C7—C8	1.4995 (19)	C13—N2	1.4543 (18)

C8—N1	1.4098 (17)	C13—H13A	0.9600
C8—O2	1.4966 (16)	C13—H13B	0.9600
C8—H8	0.9800	C13—H13C	0.9600
C9—N1	1.4629 (16)		

C1—O2—C8	110.62 (10)	O2—C8—H8	108.8
C8—N1—C12	115.28 (10)	C7—C8—H8	108.8
C8—N1—C9	116.08 (10)	N1—C9—C10	109.26 (10)
C12—N1—C9	110.46 (10)	N1—C9—H9A	109.8
C11—N2—C10	109.72 (10)	C10—C9—H9A	109.8
C11—N2—C13	110.04 (11)	N1—C9—H9B	109.8
C10—N2—C13	111.48 (12)	C10—C9—H9B	109.8
O1—C1—O2	122.13 (14)	H9A—C9—H9B	108.3
O1—C1—C2	129.07 (15)	N2—C10—C9	110.63 (11)
O2—C1—C2	108.78 (12)	N2—C10—H10A	109.5
C7—C2—C3	121.67 (14)	C9—C10—H10A	109.5
C7—C2—C1	108.45 (12)	N2—C10—H10B	109.5
C3—C2—C1	129.87 (14)	C9—C10—H10B	109.5
C4—C3—C2	117.60 (15)	H10A—C10—H10B	108.1
C4—C3—H3	121.2	N2—C11—C12	111.45 (10)
C2—C3—H3	121.2	N2—C11—H11A	109.3
C3—C4—C5	120.85 (15)	C12—C11—H11A	109.3
C3—C4—H4	119.6	N2—C11—H11B	109.3
C5—C4—H4	119.6	C12—C11—H11B	109.3
C6—C5—C4	121.35 (14)	H11A—C11—H11B	108.0
C6—C5—H5	119.3	N1—C12—C11	109.89 (10)
C4—C5—H5	119.3	N1—C12—H12A	109.7
C5—C6—C7	117.86 (14)	C11—C12—H12A	109.7
C5—C6—H6	121.1	N1—C12—H12B	109.7
C7—C6—H6	121.1	C11—C12—H12B	109.7
C2—C7—C6	120.67 (13)	H12A—C12—H12B	108.2
C2—C7—C8	109.69 (12)	N2—C13—H13A	109.5
C6—C7—C8	129.64 (13)	N2—C13—H13B	109.5
N1—C8—O2	113.53 (10)	H13A—C13—H13B	109.5
N1—C8—C7	114.27 (11)	N2—C13—H13C	109.5
O2—C8—C7	102.45 (10)	H13A—C13—H13C	109.5
N1—C8—H8	108.8	H13B—C13—H13C	109.5

O1—C1—O2—C8	−179.48 (12)	C2—C7—C8—O2	−1.01 (13)
C2—C1—O2—C8	−0.77 (13)	C6—C7—C8—O2	178.33 (12)
O1—C1—C2—C7	178.69 (14)	N1—C8—O2—C1	124.81 (11)
O2—C1—C2—C7	0.10 (14)	C7—C8—O2—C1	1.08 (13)
O1—C1—C2—C3	−0.2 (2)	O2—C8—N1—C12	−55.67 (14)
O2—C1—C2—C3	−178.84 (13)	C7—C8—N1—C12	61.34 (15)
C7—C2—C3—C4	−0.5 (2)	O2—C8—N1—C9	75.74 (14)
C1—C2—C3—C4	178.35 (13)	C7—C8—N1—C9	−167.25 (11)
C2—C3—C4—C5	0.5 (2)	C10—C9—N1—C8	167.50 (11)
C3—C4—C5—C6	−0.4 (2)	C10—C9—N1—C12	−58.87 (14)

C4—C5—C6—C7	0.1 (2)	N1—C9—C10—N2	59.53 (14)
C3—C2—C7—C6	0.25 (19)	C9—C10—N2—C11	−58.56 (13)
C1—C2—C7—C6	−178.80 (11)	C9—C10—N2—C13	179.29 (11)
C3—C2—C7—C8	179.66 (12)	C12—C11—N2—C10	57.22 (14)
C1—C2—C7—C8	0.61 (14)	C12—C11—N2—C13	−179.77 (12)
C5—C6—C7—C2	−0.08 (19)	N2—C11—C12—N1	−56.78 (14)
C5—C6—C7—C8	−179.35 (13)	C11—C12—N1—C8	−168.52 (10)
C2—C7—C8—N1	−124.24 (12)	C11—C12—N1—C9	57.45 (14)
C6—C7—C8—N1	55.10 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.98	2.69	3.6135 (18)	157
C10—H10*A*···N2^{ii}	0.97	2.60	3.5315 (17)	160

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