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[4-(4-Methoxyphenyl)-1-methyl-3-nitropyrrolidin-3-yl]methanol

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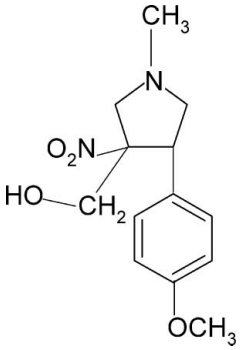
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.162; data-to-parameter ratio = 19.8.

In the title compound, $C_{13}H_{18}N_2O_4$, the dihedral angle between the benzene and pyrrolidine (all atoms) rings is 70.6 (1)°. The pyrrolidine ring adopts a half-chair conformation. In the crystal, molecules form chains along the *c*-axis direction linked by O—H···N hydrogen bonds, which are then connected by C—H···O interactions, forming a sheet parallel to the *bc* plane.

Related literature

For information on the pyrrolidine ring in biologically active natural compounds, see: Gu *et al.* (2004). For the use of pyrrolidine-containing molecules in the treatment of diseases, see, for example: Horri *et al.* (1986) for diabetes and Karpas *et al.* (1988) for viral infections. For bond lengths in a related structure, see: Jayabharathi *et al.* (2009).



Experimental

Crystal data

$C_{13}H_{18}N_2O_4$ M_r = 266.29
Monoclinic, $P2_1/c$
a = 11.6827 (10) Å
b = 11.1912 (11) Å
c = 11.1789 (11) Å
β = 109.118 (2)°
V = 1381.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
12464 measured reflections
3407 independent reflections
2282 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.162
S = 1.01
3407 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N1^{i}	0.82	2.01	2.8237 (16)	170
C1—H1A···O2^{ii}	0.96	2.51	3.390 (2)	153
C3—H3···O3^{iii}	0.93	2.51	3.429 (2)	171

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x-1, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); 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, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NK2193).

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supporting information

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S1. Comment

The stucture of the title compound, (I), is shown below. Dimensions are available in the archived CIF.

Pyrrolidine ring is present in many biologically active natural compounds and pharmaceuticals (Gu *et al.*, 2004), and find utility in the treatment of diseases such as diabetes (Horri *et al.*,1986), and viral infections (Karpas *et al.*, 1988).

The bond lengths C8—C13 = 1.525 (2) Å; C13—N1=1.460 (2) Å; C11—N1 = 1.462 (2) Å; C11—C9= 1.520 (2) Å; C8—C9= 1.566 (2) Å are longer than the normal values but are comparable with the values of such distances in the reported structure (Jayabharathi *et al.*, 2009). This may be due to the steric forces caused by the bulky group at C8 and C9 of pyrrolidine moiety. C1—O1 [1.416 (3) Å] is longer than C2—O1 [1.367 (2) Å]; this may be due the end atom C1.The dihedral angle between phenyl and pyrrolidine ring is 70.6 (1)°. The sum of angles around N3 [360°] and N1[329.1 (1)°] indicates $sp^2$and sp^3hybridization, respectively. The five membered ring adopts half chair conformation.The crystal structure is stabilized by intermolecular O—H···N and C—H···O type hydrogen bonds.

S2. Experimental

Typical Procedure for the synthesis of (E)-3-(4-methoxyphenyl)-2-nitroprop-2-en-1-ol:

To a stirred soln of (E)-1-methoxy-4-(2-nitrovinyl)benzene (10 mmol) in THF (20 mL) at r.t. was added imidazole (1 equiv) followed by anthranilic acid (10 mol%). Aq formaldehyde (38%, 20 mL, excess) was then added and the mixture was stirred at r.t. for the period of 48h. On completion of the reaction (TLC analysis), the mixture was acidified with 5 M HCl (20 mL) and the aqueous layer was extracted with EtOAc (3 × 25 mL). The combined organic layers were washed with brine (50 mL), dried (anhyd Na2SO4), and concentrated in vacuo. The residue was purified by column chromatography (silica gel, EtOAc–hexanes, 0–25%, gradient elution) to afford pure (E)-3-(4-methoxyphenyl)-2-nitro-prop-2-en-1-ol in 50% yield as yellow oil.

A mixture of (*E*)-3-(4-methoxyphenyl)-2-nitroprop-2-en-1-ol (2 mmol,0.42 g), *para* formaldehyde (12 mmol,0.36 g) and sacrosine (6 mmol,0.53 g) in acetonitrile(8 ml) was refluxed for 8hrs. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated and the resulting crude mass was diluted with water (20 ml) and extracted with ethyl acetate (3x10ml) and dried over anhydrous Na_2SO_4.The organic layer was concentrated and purified by column chromatography on silica gel (Acme 100–200 mesh), using ethyl acetate:hexane (3:7) to provide the title compound as a colourless solid in 73% (0.39 g) yield.

S3. Refinement

H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distance of 0.93 - 0.97 Å, O—H distance of 0.82 Å and U_{iso}(H) = 1.2Ueq(N,*C*).

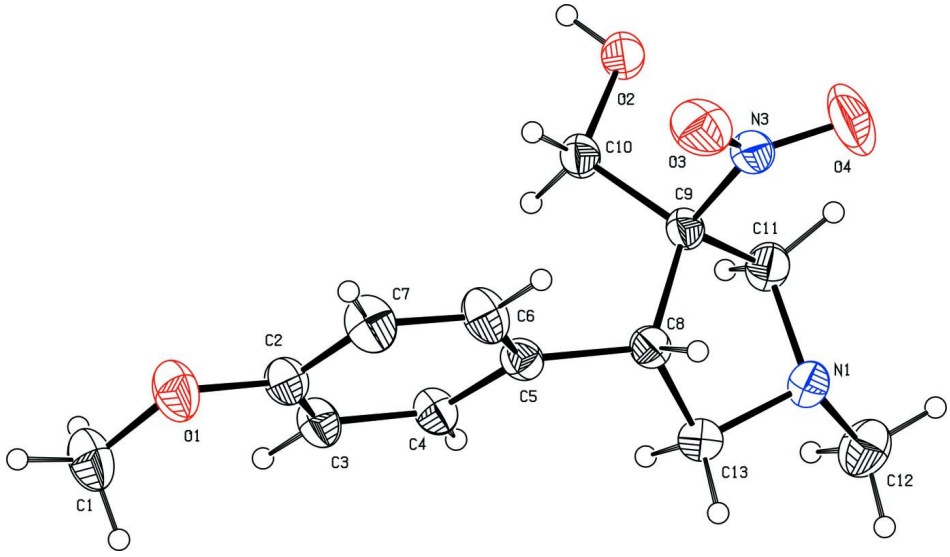

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

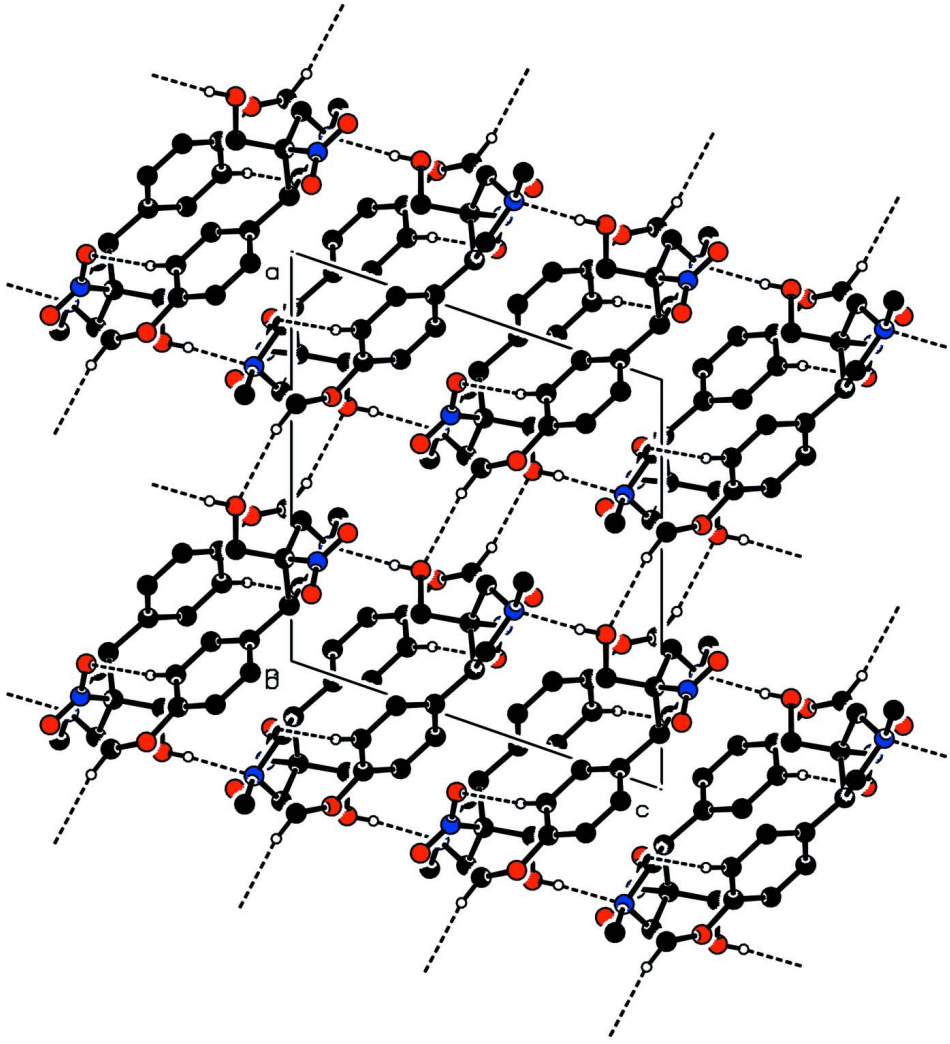

Figure 2

The packing of the molecules in the crystal structure. The dashed lines indicate the hydrogen bonds.

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Crystal data

$C_{13}H_{18}N_2O_4$
$M_r = 266.29$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.6827\ (10)$ Å
$b = 11.1912\ (11)$ Å
$c = 11.1789\ (11)$ Å
$\beta = 109.118\ (2)°$
$V = 1381.0\ (2)$ Å^3
$Z = 4$

$F(000) = 568$
$D_x = 1.281$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3407 reflections
$\theta = 1.5–28.3°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, colourless
0.22 × 0.20 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
12464 measured reflections
3407 independent reflections
2282 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.8°$
$h = -15 \rightarrow 15$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.162$
$S = 1.01$
3407 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ 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_{iso}*/U_{eq}
C1	−0.36799 (16)	0.15244 (19)	0.51274 (18)	0.0686 (5)
H1A	−0.4468	0.1772	0.4592	0.103*
H1B	−0.3743	0.0770	0.5509	0.103*
H1C	−0.3151	0.1445	0.4631	0.103*
C2	−0.20810 (13)	0.21887 (15)	0.69433 (15)	0.0481 (4)
C3	−0.13157 (13)	0.12749 (15)	0.68781 (14)	0.0487 (4)
H3	−0.1540	0.0751	0.6195	0.058*
C4	−0.02079 (13)	0.11333 (14)	0.78317 (14)	0.0451 (4)
H4	0.0301	0.0513	0.7772	0.054*
C5	0.01580 (12)	0.18913 (13)	0.88680 (13)	0.0397 (3)
C6	−0.06274 (14)	0.28269 (16)	0.88911 (16)	0.0544 (4)
H6	−0.0407	0.3355	0.9571	0.065*
C7	−0.17096 (15)	0.29922 (17)	0.79468 (17)	0.0589 (5)
H7	−0.2197	0.3641	0.7976	0.071*
C8	0.13415 (12)	0.17615 (13)	0.99434 (12)	0.0388 (3)
H8	0.1198	0.2054	1.0708	0.047*
C9	0.24446 (12)	0.24842 (13)	0.98146 (12)	0.0380 (3)

C10	0.22476 (13)	0.29756 (15)	0.84980 (13)	0.0443 (4)
H10A	0.2056	0.2330	0.7885	0.053*
H10B	0.1576	0.3535	0.8270	0.053*
C11	0.35084 (14)	0.16266 (15)	1.02668 (14)	0.0484 (4)
H11A	0.3672	0.1256	0.9555	0.058*
H11B	0.4232	0.2039	1.0783	0.058*
C12	0.38953 (16)	−0.03316 (17)	1.12644 (17)	0.0643 (5)
H12A	0.4701	−0.0119	1.1783	0.096*
H12B	0.3916	−0.0659	1.0478	0.096*
H12C	0.3573	−0.0915	1.1697	0.096*
C13	0.18708 (14)	0.05085 (15)	1.02428 (15)	0.0481 (4)
H13A	0.1451	0.0054	1.0711	0.058*
H13B	0.1826	0.0079	0.9475	0.058*
N1	0.31240 (11)	0.07359 (12)	1.10121 (11)	0.0433 (3)
N3	0.26471 (14)	0.35622 (12)	1.07021 (12)	0.0509 (4)
O1	−0.32042 (10)	0.23895 (12)	0.60855 (13)	0.0663 (4)
O2	0.33114 (10)	0.35553 (12)	0.84933 (10)	0.0578 (4)
H2	0.3213	0.3832	0.7788	0.087*
O3	0.18039 (14)	0.42471 (12)	1.05376 (14)	0.0740 (4)
O4	0.36032 (15)	0.36987 (16)	1.15196 (15)	0.1019 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0467 (9)	0.0815 (14)	0.0648 (12)	0.0034 (9)	0.0006 (8)	−0.0089 (10)
C2	0.0367 (7)	0.0534 (10)	0.0510 (9)	−0.0001 (7)	0.0101 (7)	0.0014 (7)
C3	0.0456 (8)	0.0500 (9)	0.0461 (8)	−0.0014 (7)	0.0091 (7)	−0.0101 (7)
C4	0.0396 (8)	0.0449 (9)	0.0480 (8)	0.0002 (6)	0.0107 (7)	−0.0060 (6)
C5	0.0366 (7)	0.0443 (8)	0.0403 (7)	−0.0048 (6)	0.0155 (6)	−0.0034 (6)
C6	0.0462 (9)	0.0618 (11)	0.0532 (9)	0.0018 (8)	0.0138 (7)	−0.0168 (8)
C7	0.0485 (9)	0.0589 (11)	0.0675 (11)	0.0111 (8)	0.0164 (9)	−0.0118 (8)
C8	0.0392 (7)	0.0449 (8)	0.0341 (7)	−0.0050 (6)	0.0147 (6)	−0.0028 (6)
C9	0.0385 (7)	0.0426 (8)	0.0312 (7)	−0.0060 (6)	0.0092 (6)	−0.0026 (5)
C10	0.0414 (8)	0.0549 (9)	0.0348 (7)	−0.0099 (7)	0.0098 (6)	0.0016 (6)
C11	0.0406 (8)	0.0611 (10)	0.0440 (8)	0.0002 (7)	0.0148 (7)	0.0069 (7)
C12	0.0694 (12)	0.0594 (12)	0.0626 (11)	0.0198 (9)	0.0196 (9)	0.0076 (8)
C13	0.0489 (8)	0.0451 (9)	0.0473 (8)	−0.0063 (7)	0.0116 (7)	0.0033 (6)
N1	0.0432 (7)	0.0483 (8)	0.0377 (6)	0.0036 (5)	0.0124 (5)	0.0043 (5)
N3	0.0627 (9)	0.0478 (8)	0.0384 (7)	−0.0138 (7)	0.0114 (6)	−0.0022 (6)
O1	0.0463 (7)	0.0686 (9)	0.0692 (8)	0.0104 (6)	−0.0013 (6)	−0.0085 (6)
O2	0.0480 (6)	0.0810 (9)	0.0414 (6)	−0.0216 (6)	0.0105 (5)	0.0112 (5)
O3	0.0861 (10)	0.0537 (8)	0.0824 (9)	0.0001 (7)	0.0281 (8)	−0.0176 (7)
O4	0.0916 (11)	0.0971 (12)	0.0788 (10)	−0.0172 (9)	−0.0244 (9)	−0.0309 (9)

Geometric parameters (Å, º)

C1—O1	1.415 (2)	C9—C10	1.5165 (19)
C1—H1A	0.9600	C9—C11	1.520 (2)

C1—H1B	0.9600	C9—N3	1.5303 (19)
C1—H1C	0.9600	C10—O2	1.4035 (17)
C2—O1	1.3666 (18)	C10—H10A	0.9700
C2—C3	1.376 (2)	C10—H10B	0.9700
C2—C7	1.392 (2)	C11—N1	1.4610 (19)
C3—C4	1.390 (2)	C11—H11A	0.9700
C3—H3	0.9300	C11—H11B	0.9700
C4—C5	1.386 (2)	C12—N1	1.467 (2)
C4—H4	0.9300	C12—H12A	0.9600
C5—C6	1.398 (2)	C12—H12B	0.9600
C5—C8	1.5130 (19)	C12—H12C	0.9600
C6—C7	1.369 (2)	C13—N1	1.4571 (19)
C6—H6	0.9300	C13—H13A	0.9700
C7—H7	0.9300	C13—H13B	0.9700
C8—C13	1.525 (2)	N3—O4	1.1989 (19)
C8—C9	1.5676 (18)	N3—O3	1.214 (2)
C8—H8	0.9800	O2—H2	0.8200

O1—C1—H1A	109.5	C11—C9—C8	104.53 (12)
O1—C1—H1B	109.5	N3—C9—C8	107.72 (11)
H1A—C1—H1B	109.5	O2—C10—C9	108.55 (11)
O1—C1—H1C	109.5	O2—C10—H10A	110.0
H1A—C1—H1C	109.5	C9—C10—H10A	110.0
H1B—C1—H1C	109.5	O2—C10—H10B	110.0
O1—C2—C3	125.19 (14)	C9—C10—H10B	110.0
O1—C2—C7	115.70 (14)	H10A—C10—H10B	108.4
C3—C2—C7	119.11 (14)	N1—C11—C9	104.48 (11)
C2—C3—C4	120.13 (14)	N1—C11—H11A	110.9
C2—C3—H3	119.9	C9—C11—H11A	110.9
C4—C3—H3	119.9	N1—C11—H11B	110.9
C5—C4—C3	121.74 (14)	C9—C11—H11B	110.9
C5—C4—H4	119.1	H11A—C11—H11B	108.9
C3—C4—H4	119.1	N1—C12—H12A	109.5
C4—C5—C6	116.70 (13)	N1—C12—H12B	109.5
C4—C5—C8	123.84 (13)	H12A—C12—H12B	109.5
C6—C5—C8	119.46 (13)	N1—C12—H12C	109.5
C7—C6—C5	122.22 (15)	H12A—C12—H12C	109.5
C7—C6—H6	118.9	H12B—C12—H12C	109.5
C5—C6—H6	118.9	N1—C13—C8	103.05 (12)
C6—C7—C2	119.99 (15)	N1—C13—H13A	111.2
C6—C7—H7	120.0	C8—C13—H13A	111.2
C2—C7—H7	120.0	N1—C13—H13B	111.2
C5—C8—C13	117.52 (12)	C8—C13—H13B	111.2
C5—C8—C9	116.25 (11)	H13A—C13—H13B	109.1
C13—C8—C9	102.02 (11)	C13—N1—C11	102.69 (11)
C5—C8—H8	106.8	C13—N1—C12	113.98 (14)
C13—C8—H8	106.8	C11—N1—C12	112.41 (12)
C9—C8—H8	106.8	O4—N3—O3	122.84 (16)

C10—C9—C11	113.55 (12)	O4—N3—C9	120.15 (15)
C10—C9—N3	106.59 (12)	O3—N3—C9	117.01 (13)
C11—C9—N3	110.26 (12)	C2—O1—C1	117.86 (14)
C10—C9—C8	114.09 (11)	C10—O2—H2	109.5
O1—C2—C3—C4	−177.74 (15)	C11—C9—C10—O2	−57.84 (17)
C7—C2—C3—C4	2.5 (2)	N3—C9—C10—O2	63.75 (15)
C2—C3—C4—C5	0.3 (2)	C8—C9—C10—O2	−177.49 (12)
C3—C4—C5—C6	−1.7 (2)	C10—C9—C11—N1	−144.62 (12)
C3—C4—C5—C8	178.72 (14)	N3—C9—C11—N1	95.86 (13)
C4—C5—C6—C7	0.4 (2)	C8—C9—C11—N1	−19.66 (14)
C8—C5—C6—C7	179.95 (16)	C5—C8—C13—N1	163.40 (11)
C5—C6—C7—C2	2.4 (3)	C9—C8—C13—N1	35.02 (13)
O1—C2—C7—C6	176.40 (16)	C8—C13—N1—C11	−48.96 (14)
C3—C2—C7—C6	−3.8 (3)	C8—C13—N1—C12	−170.81 (12)
C4—C5—C8—C13	−28.9 (2)	C9—C11—N1—C13	42.62 (14)
C6—C5—C8—C13	151.54 (15)	C9—C11—N1—C12	165.54 (13)
C4—C5—C8—C9	92.35 (17)	C10—C9—N3—O4	−115.56 (17)
C6—C5—C8—C9	−87.21 (17)	C11—C9—N3—O4	8.10 (19)
C5—C8—C9—C10	−13.76 (18)	C8—C9—N3—O4	121.60 (16)
C13—C8—C9—C10	115.42 (13)	C10—C9—N3—O3	64.81 (17)
C5—C8—C9—C11	−138.37 (12)	C11—C9—N3—O3	−171.52 (13)
C13—C8—C9—C11	−9.19 (13)	C8—C9—N3—O3	−58.03 (17)
C5—C8—C9—N3	104.35 (13)	C3—C2—O1—C1	7.2 (3)
C13—C8—C9—N3	−126.47 (12)	C7—C2—O1—C1	−173.08 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1^{i}	0.82	2.01	2.8237 (16)	170
C1—H1*A*···O2^{ii}	0.96	2.51	3.390 (2)	153
C3—H3···O3^{iii}	0.93	2.51	3.429 (2)	171

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) *x*−1, −*y*+1/2, *z*−1/2; (iii) −*x*, *y*−1/2, −*z*+3/2.