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(Morpholin-4-yl)[2-(morpholin-4-yl)-3,5-dinitrophenyl]methanone

Chao Gao, Ying Xiong, Yong Xia and Luo-Ting Yu*

State Key Laboratory of Biotherapy and Cancer Center, West China Hospital, West China Medical School, Sichuan University, Chengdu 610041, People's Republic of China
Correspondence e-mail: yuluot@scu.edu.cn

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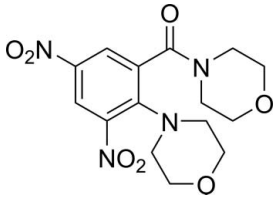
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.110; data-to-parameter ratio = 14.6.

In the title compound, $C_{15}H_{18}N_4O_7$, the morpholine rings adopt chair conformations. The benzene ring forms dihedral angles of 55.94 (7) and 63.19 (7)° with the planes through the C atoms of the two morpholine rings.

Related literature

For the biological activity of benzamide derivatives, see: Christophe *et al.* (2009).



Experimental

Crystal data

$C_{15}H_{18}N_4O_7$
M_r = 366.33
Monoclinic, $P2_1/c$
a = 10.2640 (4) Å
b = 21.5488 (7) Å
c = 8.0061 (3) Å
β = 108.587 (4)°
V = 1678.40 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.38 × 0.35 × 0.35 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2006)
T_{min} = 0.994, T_{max} = 1.000
7068 measured reflections
3422 independent reflections
2440 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.110
S = 1.03
3422 reflections
235 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2006); 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: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

We thank the Analytical and Testing Center of Sichuan University for the X-ray measurements.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2263).

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supporting information

Acta Cryst. (2012). E**68**, o376 [doi:10.1107/S1600536812000426]

(Morpholin-4-yl)[2-(morpholin-4-yl)-3,5-dinitrophenyl]methanone

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S1. Comment

Benzamide derivatives are of great importance owing to their antibacterial properties (Christophe *et al.*, 2009). The title compound is one of the key intermediates in our synthetic investigations of antibacterial drugs. Therefore, its crystal structure was determined.

In the crystal structure of the title compound, $C_{15}H_{18}N_4O_7$, the morpholine rings adopt a chair conformation. The benzene ring forms dihedral angles of 55.94 (7)° and 63.19 (7)° with the two morpholine rings.

S2. Experimental

3.42 g (12.9 mmol) of 2-chloro-3,5-dinitrobenzoyl chloride was added to a solution of 2.36 g (27.1 mmol) morpholine in 20 ml of dichloromethane and 1.82 g (17.9 mmol) triethylamine. The mixture was stirred for 2 h at room temperature extracted with water and dichloromethane and the organic solvent was evaporated and the title compound was recrystallized from ethanol (yield 4.25 g, 90%). Crystals suitable for X-ray analysis were obtained by slow evaporation of the solvent from a solution of the title compound in acetonitrile.

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

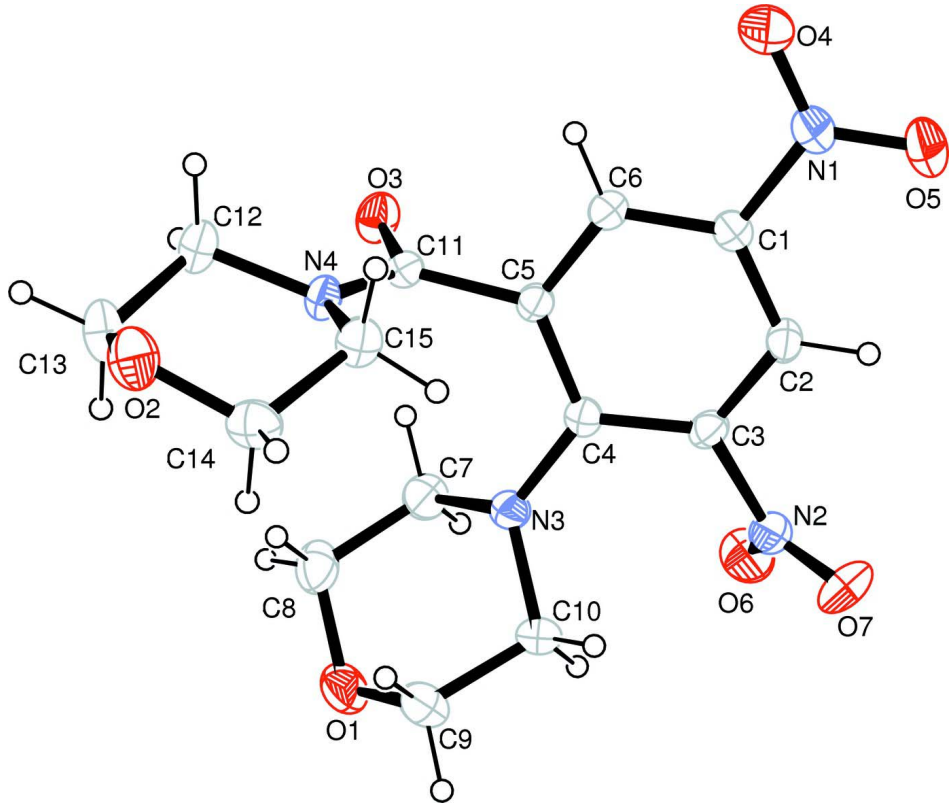

Figure 1

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

(Morpholin-4-yl)[2-(morpholin-4-yl)-3,5-dinitrophenyl]methanone

Crystal data

$C_{15}H_{18}N_4O_7$
$M_r = 366.33$
Monoclinic, $P2_1/c$
$a = 10.2640$ (4) Å
$b = 21.5488$ (7) Å
$c = 8.0061$ (3) Å
$\beta = 108.587$ (4)°
$V = 1678.40$ (10) Å^3
$Z = 4$
$F(000) = 768$
$D_x = 1.450$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 2613 reflections
$\theta = 3.0$–$29.0°$
$\mu = 0.12$ mm^{-1}
$T = 293$ K
Block, yellow
0.38 × 0.35 × 0.35 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0874 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2006)
$T_{min} = 0.994$, $T_{max} = 1.000$
7068 measured reflections
3422 independent reflections
2440 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.0°$
$h = -12 \rightarrow 7$
$k = -26 \rightarrow 18$
$l = -9 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.110$
$S = 1.03$
3422 reflections
235 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
$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.3019P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Experimental. CrysAlisPro (Oxford Diffraction, 2006). Agilent Technologies, Version 1.171.35.19 (release 27-10-2011 CrysAlis171 .NET) (compiled Oct 27 2011,15:02:11) 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.38171 (14)	0.03743 (7)	0.58276 (17)	0.0608 (4)
O2	0.58939 (14)	0.11924 (7)	0.02632 (18)	0.0588 (4)
O3	0.27069 (12)	0.25026 (6)	0.21720 (17)	0.0468 (3)
O4	−0.13992 (17)	0.22884 (9)	−0.4060 (2)	0.0884 (6)
O5	−0.30029 (15)	0.16911 (8)	−0.38749 (19)	0.0723 (5)
O6	−0.11367 (15)	0.07133 (8)	0.3247 (2)	0.0697 (5)
O7	−0.17948 (18)	0.01161 (7)	0.0962 (2)	0.0825 (5)
N1	−0.18359 (16)	0.18932 (8)	−0.3309 (2)	0.0500 (4)
N2	−0.12006 (16)	0.05713 (8)	0.1748 (2)	0.0510 (4)
N3	0.17106 (14)	0.09335 (6)	0.30645 (17)	0.0375 (4)
N4	0.36454 (13)	0.17369 (6)	0.09991 (17)	0.0342 (3)
C1	−0.09372 (17)	0.16569 (8)	−0.1628 (2)	0.0361 (4)
C2	−0.14151 (17)	0.12014 (8)	−0.0794 (2)	0.0373 (4)
H2	−0.2291	0.1036	−0.1289	0.045*
C3	−0.05571 (17)	0.09943 (8)	0.0810 (2)	0.0342 (4)
C4	0.07972 (16)	0.12074 (7)	0.1600 (2)	0.0307 (4)
C5	0.12146 (16)	0.16958 (7)	0.0703 (2)	0.0306 (4)
C6	0.03563 (17)	0.19131 (8)	−0.0891 (2)	0.0357 (4)
H6	0.0648	0.2232	−0.1471	0.043*
C7	0.25205 (19)	0.12947 (9)	0.4592 (2)	0.0464 (5)
H7A	0.2059	0.1302	0.5476	0.056*
H7B	0.2613	0.1719	0.4238	0.056*

C8	0.3922 (2)	0.10043 (11)	0.5348 (3)	0.0563 (6)
H8A	0.4402	0.1024	0.4485	0.068*
H8B	0.4451	0.1236	0.6379	0.068*
C9	0.3045 (2)	0.00286 (10)	0.4328 (3)	0.0570 (5)
H9A	0.2990	−0.0400	0.4668	0.068*
H9B	0.3515	0.0037	0.3452	0.068*
C10	0.16127 (19)	0.02827 (8)	0.3529 (3)	0.0460 (5)
H10A	0.1125	0.0047	0.2485	0.055*
H10B	0.1110	0.0249	0.4366	0.055*
C11	0.25990 (16)	0.20097 (8)	0.1370 (2)	0.0317 (4)
C12	0.49371 (17)	0.20777 (9)	0.1316 (3)	0.0456 (5)
H12A	0.5083	0.2348	0.2329	0.055*
H12B	0.4886	0.2334	0.0301	0.055*
C13	0.61214 (19)	0.16320 (11)	0.1647 (3)	0.0594 (6)
H13A	0.6957	0.1862	0.1754	0.071*
H13B	0.6246	0.1416	0.2749	0.071*
C14	0.4726 (2)	0.08302 (9)	0.0182 (3)	0.0493 (5)
H14A	0.4871	0.0625	0.1305	0.059*
H14B	0.4603	0.0513	−0.0714	0.059*
C15	0.34554 (18)	0.12219 (8)	−0.0241 (2)	0.0419 (4)
H15A	0.3240	0.1383	−0.1429	0.050*
H15B	0.2688	0.0968	−0.0189	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0474 (8)	0.0784 (11)	0.0501 (8)	0.0055 (7)	0.0066 (7)	0.0204 (8)
O2	0.0462 (8)	0.0699 (10)	0.0680 (9)	0.0094 (7)	0.0290 (7)	−0.0044 (8)
O3	0.0433 (7)	0.0360 (7)	0.0618 (8)	−0.0055 (6)	0.0177 (6)	−0.0154 (6)
O4	0.0654 (11)	0.1098 (14)	0.0752 (11)	−0.0167 (10)	0.0017 (9)	0.0537 (11)
O5	0.0423 (8)	0.0936 (12)	0.0631 (9)	−0.0114 (8)	−0.0086 (7)	0.0163 (8)
O6	0.0582 (10)	0.0991 (13)	0.0616 (10)	0.0069 (8)	0.0327 (8)	0.0293 (9)
O7	0.0735 (11)	0.0540 (10)	0.1152 (14)	−0.0298 (9)	0.0236 (10)	0.0129 (10)
N1	0.0393 (9)	0.0576 (11)	0.0462 (9)	−0.0007 (8)	0.0038 (7)	0.0108 (8)
N2	0.0357 (9)	0.0519 (11)	0.0666 (11)	0.0001 (8)	0.0183 (8)	0.0201 (9)
N3	0.0382 (8)	0.0352 (8)	0.0352 (7)	−0.0014 (6)	0.0062 (6)	0.0034 (6)
N4	0.0290 (7)	0.0343 (8)	0.0393 (8)	−0.0048 (6)	0.0111 (6)	−0.0061 (6)
C1	0.0323 (9)	0.0381 (10)	0.0361 (9)	0.0012 (7)	0.0084 (7)	0.0040 (8)
C2	0.0268 (8)	0.0387 (10)	0.0446 (10)	−0.0034 (7)	0.0090 (7)	−0.0009 (8)
C3	0.0336 (9)	0.0305 (9)	0.0423 (9)	−0.0025 (7)	0.0176 (8)	0.0024 (8)
C4	0.0300 (8)	0.0306 (9)	0.0325 (8)	0.0006 (7)	0.0114 (7)	−0.0032 (7)
C5	0.0295 (8)	0.0279 (8)	0.0356 (8)	−0.0003 (7)	0.0122 (7)	−0.0014 (7)
C6	0.0332 (9)	0.0343 (9)	0.0406 (9)	−0.0020 (7)	0.0131 (7)	0.0048 (8)
C7	0.0502 (11)	0.0525 (12)	0.0341 (9)	−0.0053 (9)	0.0100 (8)	−0.0001 (8)
C8	0.0444 (11)	0.0781 (16)	0.0420 (11)	−0.0116 (11)	0.0076 (9)	0.0061 (11)
C9	0.0494 (12)	0.0536 (13)	0.0646 (13)	0.0120 (10)	0.0134 (10)	0.0161 (11)
C10	0.0458 (11)	0.0374 (10)	0.0510 (11)	0.0012 (8)	0.0102 (9)	0.0137 (9)
C11	0.0320 (9)	0.0291 (9)	0.0334 (8)	−0.0023 (7)	0.0097 (7)	0.0018 (7)

C12	0.0339 (10)	0.0499 (12)	0.0546 (11)	−0.0099 (8)	0.0163 (8)	−0.0076 (9)
C13	0.0320 (10)	0.0768 (16)	0.0670 (13)	0.0005 (10)	0.0125 (10)	−0.0057 (12)
C14	0.0572 (13)	0.0440 (11)	0.0521 (11)	0.0086 (10)	0.0252 (10)	0.0016 (9)
C15	0.0421 (10)	0.0372 (10)	0.0477 (10)	−0.0012 (8)	0.0164 (8)	−0.0102 (8)

Geometric parameters (Å, º)

O1—C8	1.424 (2)	C5—C6	1.381 (2)
O1—C9	1.420 (2)	C5—C11	1.509 (2)
O2—C13	1.419 (2)	C6—H6	0.9300
O2—C14	1.414 (2)	C7—H7A	0.9700
O3—C11	1.2275 (19)	C7—H7B	0.9700
O4—N1	1.208 (2)	C7—C8	1.507 (3)
O5—N1	1.218 (2)	C8—H8A	0.9700
O6—N2	1.220 (2)	C8—H8B	0.9700
O7—N2	1.219 (2)	C9—H9A	0.9700
N1—C1	1.460 (2)	C9—H9B	0.9700
N2—C3	1.465 (2)	C9—C10	1.507 (2)
N3—C4	1.379 (2)	C10—H10A	0.9700
N3—C7	1.464 (2)	C10—H10B	0.9700
N3—C10	1.463 (2)	C12—H12A	0.9700
N4—C11	1.338 (2)	C12—H12B	0.9700
N4—C12	1.465 (2)	C12—C13	1.505 (3)
N4—C15	1.460 (2)	C13—H13A	0.9700
C1—C2	1.363 (2)	C13—H13B	0.9700
C1—C6	1.384 (2)	C14—H14A	0.9700
C2—H2	0.9300	C14—H14B	0.9700
C2—C3	1.379 (2)	C14—C15	1.499 (2)
C3—C4	1.408 (2)	C15—H15A	0.9700
C4—C5	1.415 (2)	C15—H15B	0.9700
C9—O1—C8	109.99 (14)	C7—C8—H8B	109.4
C14—O2—C13	109.29 (14)	H8A—C8—H8B	108.0
O4—N1—O5	122.95 (17)	O1—C9—H9A	109.2
O4—N1—C1	118.63 (16)	O1—C9—H9B	109.2
O5—N1—C1	118.41 (16)	O1—C9—C10	111.99 (17)
O6—N2—C3	117.18 (17)	H9A—C9—H9B	107.9
O7—N2—O6	124.81 (18)	C10—C9—H9A	109.2
O7—N2—C3	117.97 (18)	C10—C9—H9B	109.2
C4—N3—C7	122.26 (14)	N3—C10—C9	108.64 (15)
C4—N3—C10	122.91 (14)	N3—C10—H10A	110.0
C10—N3—C7	111.36 (14)	N3—C10—H10B	110.0
C11—N4—C12	119.16 (14)	C9—C10—H10A	110.0
C11—N4—C15	122.84 (13)	C9—C10—H10B	110.0
C15—N4—C12	114.60 (13)	H10A—C10—H10B	108.3
C2—C1—N1	118.85 (15)	O3—C11—N4	123.45 (15)
C2—C1—C6	121.64 (15)	O3—C11—C5	119.39 (15)
C6—C1—N1	119.47 (15)	N4—C11—C5	117.13 (14)

C1—C2—H2	121.1	N4—C12—H12A	109.6
C1—C2—C3	117.78 (15)	N4—C12—H12B	109.6
C3—C2—H2	121.1	N4—C12—C13	110.25 (16)
C2—C3—N2	114.83 (15)	H12A—C12—H12B	108.1
C2—C3—C4	123.97 (15)	C13—C12—H12A	109.6
C4—C3—N2	120.94 (15)	C13—C12—H12B	109.6
N3—C4—C3	123.28 (15)	O2—C13—C12	111.48 (16)
N3—C4—C5	121.07 (14)	O2—C13—H13A	109.3
C3—C4—C5	115.44 (14)	O2—C13—H13B	109.3
C4—C5—C11	123.82 (14)	C12—C13—H13A	109.3
C6—C5—C4	120.93 (14)	C12—C13—H13B	109.3
C6—C5—C11	115.25 (14)	H13A—C13—H13B	108.0
C1—C6—H6	120.0	O2—C14—H14A	109.4
C5—C6—C1	120.07 (15)	O2—C14—H14B	109.4
C5—C6—H6	120.0	O2—C14—C15	111.31 (16)
N3—C7—H7A	109.8	H14A—C14—H14B	108.0
N3—C7—H7B	109.8	C15—C14—H14A	109.4
N3—C7—C8	109.28 (16)	C15—C14—H14B	109.4
H7A—C7—H7B	108.3	N4—C15—C14	110.89 (15)
C8—C7—H7A	109.8	N4—C15—H15A	109.5
C8—C7—H7B	109.8	N4—C15—H15B	109.5
O1—C8—C7	111.10 (16)	C14—C15—H15A	109.5
O1—C8—H8A	109.4	C14—C15—H15B	109.5
O1—C8—H8B	109.4	H15A—C15—H15B	108.0
C7—C8—H8A	109.4		

O1—C9—C10—N3	57.5 (2)	C4—N3—C10—C9	144.78 (16)
O2—C14—C15—N4	−54.3 (2)	C4—C5—C6—C1	0.6 (2)
O4—N1—C1—C2	−178.60 (18)	C4—C5—C11—O3	−97.7 (2)
O4—N1—C1—C6	3.5 (3)	C4—C5—C11—N4	84.29 (19)
O5—N1—C1—C2	2.8 (3)	C6—C1—C2—C3	−0.9 (3)
O5—N1—C1—C6	−175.07 (18)	C6—C5—C11—O3	83.08 (19)
O6—N2—C3—C2	−124.82 (18)	C6—C5—C11—N4	−94.98 (18)
O6—N2—C3—C4	49.6 (2)	C7—N3—C4—C3	−131.89 (17)
O7—N2—C3—C2	52.9 (2)	C7—N3—C4—C5	53.6 (2)
O7—N2—C3—C4	−132.64 (19)	C7—N3—C10—C9	−55.9 (2)
N1—C1—C2—C3	−178.74 (15)	C8—O1—C9—C10	−59.5 (2)
N1—C1—C6—C5	179.70 (15)	C9—O1—C8—C7	59.2 (2)
N2—C3—C4—N3	16.1 (2)	C10—N3—C4—C3	25.3 (2)
N2—C3—C4—C5	−169.09 (15)	C10—N3—C4—C5	−149.27 (16)
N3—C4—C5—C6	171.26 (15)	C10—N3—C7—C8	56.40 (19)
N3—C4—C5—C11	−8.0 (2)	C11—N4—C12—C13	153.48 (16)
N3—C7—C8—O1	−57.6 (2)	C11—N4—C15—C14	−154.09 (16)
N4—C12—C13—O2	54.2 (2)	C11—C5—C6—C1	179.86 (15)
C1—C2—C3—N2	171.64 (16)	C12—N4—C11—O3	−9.5 (2)
C1—C2—C3—C4	−2.6 (3)	C12—N4—C11—C5	168.46 (14)
C2—C1—C6—C5	1.9 (3)	C12—N4—C15—C14	47.0 (2)
C2—C3—C4—N3	−170.01 (15)	C13—O2—C14—C15	62.6 (2)

C2—C3—C4—C5	4.8 (2)	C14—O2—C13—C12	−62.8 (2)
C3—C4—C5—C6	−3.7 (2)	C15—N4—C11—O3	−167.52 (16)
C3—C4—C5—C11	177.09 (14)	C15—N4—C11—C5	10.4 (2)
C4—N3—C7—C8	−144.08 (16)	C15—N4—C12—C13	−46.8 (2)