Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[2-(2-Oxo-1,3-oxazolidin-3-yl)ethyl]-4-phenyl-1*H*-1,5-benzodiazepin-2(3*H*)-one

Daouda Ballo,[a] Noureddine Hamou Ahabchane,[a] Hafid Zouihri,[b] El Mokhtar Essassi[a] and Seik Weng Ng[c]*

[a]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, [b]CNRST Division UATRS, Angle Allal Fassi/FAR, BP 8027 Hay Riad, Rabat, Morocco, and [c]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

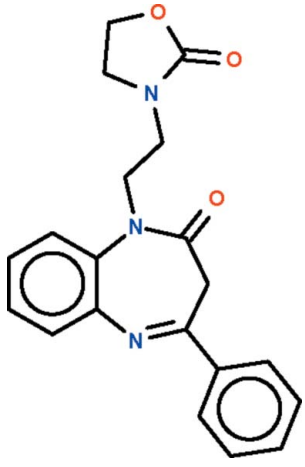
Received 7 July 2010; accepted 15 July 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.036; wR factor = 0.102; data-to-parameter ratio = 8.7.

The seven-membered ring in the title compound, $C_{20}H_{19}N_3O_3$, adopts a boat conformation with the two phenylene C atoms representing the stern and the methylene C atom the prow. The dihedral angle between the best plane through the seven-membered ring (r.m.s deviation = 0.358 Å) and the phenyl substituent is 55.8 (1)°. The two rings at either ends of the ethyl chain are staggered [N—CH_2—CH_2—N torsion angle = 57.5 (4)°].

Related literature

For the background to 2,3-dihydro-1*H*-1,5-benzodiazepin-2-ones, see: Ahabchane *et al.* (1999). For a related structure, see: Ballo *et al.* (2010).



Experimental

Crystal data

$C_{20}H_{19}N_3O_3$
M_r = 349.38
Orthorhombic, $P2_12_12_1$
a = 9.0163 (5) Å
b = 11.6671 (6) Å
c = 16.2019 (8) Å
V = 1704.34 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.25 × 0.25 × 0.15 mm

Data collection

Bruker X8 APEXII diffractometer
9253 measured reflections
2053 independent reflections
1578 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 0.90
2053 reflections
235 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2052).

References

Ahabchane, N. H., Keita, A. & Essassi, E. M. (1999). *C. R. Ser. IIC*, **2**, 519–523.
Ballo, D., Ahabchane, N. H., Zouihri, H., Essassi, E. M. & Ng, S. W. (2010). *Acta Cryst.* E**66**, o1277.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2010). E**66**, o2080 [**doi:10.1107/S160053681002828X**]

1-[2-(2-Oxo-1,3-oxazolidin-3-yl)ethyl]-4-phenyl-1*H*-1,5-benzodiazepin-2(3*H*)-one

D. Ballo, N. H. Ahabchane, H. Zouihri, E. M. Essassi and S. W. Ng

Comment

The background to the class of 2,3-dihydro-1*H*-1,5-benzodiazepin-2-ones is given in an earlier report (Ahabchane *et al.*, 1999). A recent study presented the crystal structure of 1-allyl-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-2-one (Ballo *et al.*, 2010). The present study has an oxazolidin-2-onyl-ethyl group in place of the allyl group (Scheme I, Fig. 1). The principal feature is the seven-membered ring that is fused to a phenylene ring and adopts a boat-shaped conformation, two phenylene carbons representing the stern and the methylene carbon atom the prow [r.m.s deviation 0.358 Å]. The methyl carbon deviates by 0.637 Å from the best plane. The two rings at either end of the ethyl chain are staggered [N–CH_2–CH_2–N torsion angle, 57.5 (4)°].

Experimental

To a solution of 4-phenyl-1*H*-1,5-benzodiazepin-2-one (2 g, 8,.4 mmol) in DMF (40 ml) was added dichloroethylamine hydrochloride (0.9 g, 8.4 mmol), potassium carbonate (3 g, 22.2 mmol) and a catalytic quantity of tetra-*n*-butylammonium bromide. The mixture was heated on a sand bath, the reaction monotired by thin layer chromatography. On completion of the reaction, the solvent was evaporated under reduced pressure. The residue was recrystallized from ethanol to afford the title compound as colorless crystals.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2–1.5U_{eq}(C). 1486 Friedel pairs were merged in the final cycles of the refinement.

Figures

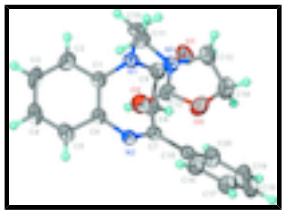


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of the molecule of $C_{20}H_{19}N_3O_3$ at the 50% probability level.

1-[2-(2-Oxo-1,3-oxazolidin-3-yl)ethyl]-4-phenyl-1*H*- 1,5-benzodiazepin-2(3*H*)-one

Crystal data

$C_{20}H_{19}N_3O_3$ $F(000) = 736$

M_r = 349.38	D_x = 1.362 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 2227 reflections
a = 9.0163 (5) Å	θ = 2.9–21.0°
b = 11.6671 (6) Å	μ = 0.09 mm^{-1}
c = 16.2019 (8) Å	T = 293 K
V = 1704.34 (15) Å^3	Prism, colorless
Z = 4	0.25 × 0.25 × 0.15 mm

Data collection

Bruker X8 APEXII diffractometer	1578 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.039
graphite	θ_{max} = 26.7°, θ_{min} = 2.9°
φ and ω scans	h = −11→10
9253 measured reflections	k = −14→13
2053 independent reflections	l = −20→14

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.036	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.102	H-atom parameters constrained
S = 0.90	$w = 1/[\sigma^2(F_o^2) + (0.0742P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
2053 reflections	$(\Delta/\sigma)_{max}$ = 0.001
235 parameters	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.3905 (2)	0.62919 (16)	0.33575 (12)	0.0439 (5)
N2	0.1611 (2)	0.45822 (16)	0.37488 (12)	0.0416 (5)
N3	0.4287 (2)	0.61202 (17)	0.51545 (12)	0.0458 (5)
O1	0.5951 (2)	0.51620 (17)	0.32572 (12)	0.0669 (5)
O2	0.1947 (2)	0.60716 (17)	0.57036 (13)	0.0677 (6)
O3	0.3700 (2)	0.48444 (16)	0.61065 (11)	0.0629 (5)
C1	0.2372 (3)	0.6471 (2)	0.31906 (14)	0.0434 (5)
C2	0.1935 (3)	0.7508 (2)	0.28491 (17)	0.0597 (7)
H2	0.2650	0.8049	0.2710	0.072*
C3	0.0461 (4)	0.7750 (2)	0.27117 (19)	0.0697 (8)
H3	0.0181	0.8445	0.2477	0.084*

C4	−0.0596 (3)	0.6950 (3)	0.29269 (17)	0.0646 (8)
H4	−0.1594	0.7110	0.2841	0.078*
C5	−0.0192 (3)	0.5927 (2)	0.32647 (15)	0.0520 (6)
H5	−0.0921	0.5403	0.3414	0.062*
C6	0.1301 (3)	0.5651 (2)	0.33911 (13)	0.0416 (5)
C7	0.2709 (3)	0.39924 (19)	0.34851 (13)	0.0405 (5)
C8	0.3658 (3)	0.4400 (2)	0.27737 (14)	0.0477 (6)
H8A	0.4248	0.3775	0.2554	0.057*
H8B	0.3046	0.4710	0.2335	0.057*
C9	0.4635 (3)	0.5312 (2)	0.31346 (14)	0.0467 (6)
C10	0.4696 (3)	0.7153 (2)	0.38443 (15)	0.0516 (6)
H10A	0.4560	0.7899	0.3592	0.062*
H10B	0.5748	0.6979	0.3839	0.062*
C11	0.4161 (3)	0.7200 (2)	0.47313 (15)	0.0483 (6)
H11A	0.4732	0.7772	0.5027	0.058*
H11B	0.3131	0.7440	0.4737	0.058*
C12	0.3193 (3)	0.5726 (2)	0.56469 (15)	0.0480 (6)
C13	0.5683 (3)	0.5585 (3)	0.53446 (17)	0.0590 (7)
H13A	0.6327	0.6096	0.5652	0.071*
H13B	0.6190	0.5333	0.4849	0.071*
C14	0.5192 (4)	0.4584 (3)	0.58632 (19)	0.0697 (8)
H14A	0.5224	0.3879	0.5546	0.084*
H14B	0.5826	0.4500	0.6343	0.084*
C15	0.3119 (3)	0.29286 (18)	0.39276 (14)	0.0415 (5)
C16	0.2634 (3)	0.2771 (2)	0.47373 (17)	0.0542 (6)
H16	0.2040	0.3321	0.4989	0.065*
C17	0.3038 (4)	0.1793 (3)	0.51664 (19)	0.0674 (8)
H17	0.2710	0.1690	0.5706	0.081*
C18	0.3914 (3)	0.0977 (3)	0.4807 (2)	0.0658 (8)
H18	0.4195	0.0331	0.5104	0.079*
C19	0.4373 (4)	0.1114 (3)	0.4010 (2)	0.0701 (8)
H19	0.4946	0.0551	0.3759	0.084*
C20	0.3988 (3)	0.2088 (2)	0.35785 (18)	0.0604 (7)
H20	0.4323	0.2180	0.3040	0.072*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0423 (11)	0.0491 (11)	0.0402 (10)	−0.0064 (9)	−0.0003 (9)	0.0029 (9)
N2	0.0395 (11)	0.0438 (10)	0.0415 (10)	−0.0009 (9)	−0.0001 (8)	−0.0040 (9)
N3	0.0414 (11)	0.0531 (11)	0.0428 (11)	0.0005 (9)	0.0037 (9)	0.0065 (9)
O1	0.0407 (10)	0.0837 (13)	0.0761 (13)	0.0036 (10)	0.0055 (9)	0.0061 (12)
O2	0.0511 (11)	0.0706 (12)	0.0815 (13)	0.0082 (11)	0.0214 (10)	0.0049 (10)
O3	0.0706 (12)	0.0607 (11)	0.0575 (11)	0.0065 (10)	0.0181 (10)	0.0150 (9)
C1	0.0470 (13)	0.0482 (13)	0.0351 (12)	0.0017 (11)	−0.0029 (11)	−0.0027 (11)
C2	0.0711 (18)	0.0510 (14)	0.0570 (16)	0.0016 (14)	−0.0068 (15)	0.0076 (12)
C3	0.080 (2)	0.0593 (16)	0.0695 (19)	0.0250 (16)	−0.0144 (17)	0.0092 (15)
C4	0.0580 (17)	0.0736 (18)	0.0623 (17)	0.0226 (16)	−0.0112 (15)	−0.0051 (15)

C5	0.0434 (13)	0.0644 (16)	0.0483 (14)	0.0065 (12)	−0.0018 (11)	−0.0093 (13)
C6	0.0447 (12)	0.0465 (13)	0.0335 (11)	0.0032 (10)	−0.0008 (10)	−0.0061 (10)
C7	0.0412 (13)	0.0444 (11)	0.0358 (11)	−0.0024 (10)	−0.0031 (10)	−0.0058 (10)
C8	0.0558 (14)	0.0542 (13)	0.0330 (11)	0.0057 (12)	0.0038 (11)	−0.0049 (11)
C9	0.0436 (14)	0.0599 (14)	0.0367 (12)	0.0005 (12)	0.0069 (11)	0.0080 (11)
C10	0.0551 (14)	0.0547 (13)	0.0451 (13)	−0.0174 (12)	−0.0012 (12)	0.0062 (11)
C11	0.0559 (15)	0.0455 (13)	0.0435 (13)	−0.0049 (11)	0.0009 (12)	0.0003 (10)
C12	0.0544 (15)	0.0460 (13)	0.0434 (13)	0.0008 (12)	0.0077 (11)	−0.0045 (11)
C13	0.0464 (14)	0.0794 (19)	0.0513 (15)	0.0070 (13)	0.0019 (12)	0.0094 (14)
C14	0.0655 (18)	0.0788 (19)	0.0650 (17)	0.0178 (16)	0.0061 (15)	0.0164 (16)
C15	0.0388 (11)	0.0417 (11)	0.0440 (13)	−0.0032 (10)	−0.0055 (10)	−0.0049 (10)
C16	0.0557 (15)	0.0557 (14)	0.0512 (15)	−0.0024 (12)	−0.0012 (13)	0.0025 (13)
C17	0.0756 (19)	0.0734 (18)	0.0532 (16)	−0.0086 (17)	−0.0077 (15)	0.0144 (15)
C18	0.0613 (17)	0.0592 (17)	0.077 (2)	0.0008 (14)	−0.0237 (16)	0.0153 (15)
C19	0.074 (2)	0.0600 (17)	0.077 (2)	0.0218 (15)	−0.0054 (17)	−0.0025 (15)
C20	0.0643 (18)	0.0614 (16)	0.0554 (16)	0.0138 (14)	−0.0014 (14)	−0.0048 (13)

Geometric parameters (Å, °)

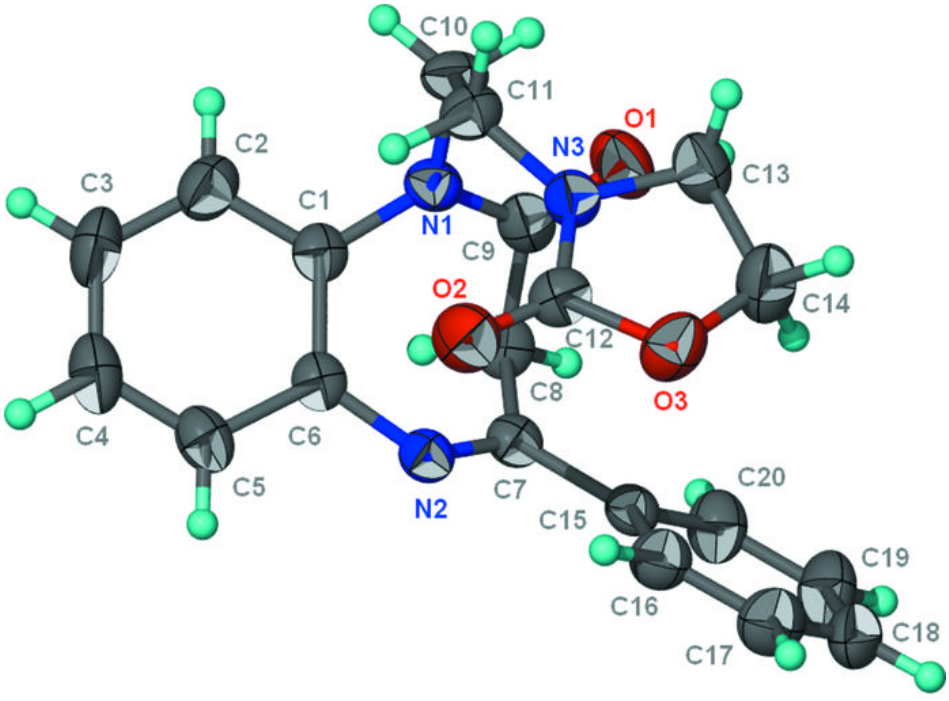
N1—C9	1.367 (3)	C8—C9	1.501 (3)
N1—C1	1.424 (3)	C8—H8A	0.9700
N1—C10	1.463 (3)	C8—H8B	0.9700
N2—C7	1.280 (3)	C10—C11	1.517 (4)
N2—C6	1.403 (3)	C10—H10A	0.9700
N3—C12	1.350 (3)	C10—H10B	0.9700
N3—C11	1.439 (3)	C11—H11A	0.9700
N3—C13	1.439 (3)	C11—H11B	0.9700
O1—C9	1.216 (3)	C13—C14	1.505 (4)
O2—C12	1.197 (3)	C13—H13A	0.9700
O3—C12	1.350 (3)	C13—H13B	0.9700
O3—C14	1.435 (4)	C14—H14A	0.9700
C1—C2	1.387 (3)	C14—H14B	0.9700
C1—C6	1.398 (3)	C15—C20	1.377 (3)
C2—C3	1.377 (4)	C15—C16	1.395 (4)
C2—H2	0.9300	C16—C17	1.385 (4)
C3—C4	1.378 (4)	C16—H16	0.9300
C3—H3	0.9300	C17—C18	1.367 (4)
C4—C5	1.363 (4)	C17—H17	0.9300
C4—H4	0.9300	C18—C19	1.364 (5)
C5—C6	1.399 (3)	C18—H18	0.9300
C5—H5	0.9300	C19—C20	1.379 (4)
C7—C15	1.480 (3)	C19—H19	0.9300
C7—C8	1.512 (3)	C20—H20	0.9300
C9—N1—C1	122.7 (2)	C11—C10—H10B	109.1
C9—N1—C10	118.8 (2)	H10A—C10—H10B	107.9
C1—N1—C10	118.3 (2)	N3—C11—C10	113.2 (2)
C7—N2—C6	119.6 (2)	N3—C11—H11A	108.9
C12—N3—C11	121.5 (2)	C10—C11—H11A	108.9
C12—N3—C13	111.40 (19)	N3—C11—H11B	108.9

C11—N3—C13	123.5 (2)	C10—C11—H11B	108.9
C12—O3—C14	109.1 (2)	H11A—C11—H11B	107.7
C2—C1—C6	119.6 (2)	O2—C12—O3	122.2 (2)
C2—C1—N1	118.7 (2)	O2—C12—N3	128.1 (2)
C6—C1—N1	121.7 (2)	O3—C12—N3	109.8 (2)
C3—C2—C1	121.2 (3)	N3—C13—C14	101.5 (2)
C3—C2—H2	119.4	N3—C13—H13A	111.5
C1—C2—H2	119.4	C14—C13—H13A	111.5
C2—C3—C4	119.1 (3)	N3—C13—H13B	111.5
C2—C3—H3	120.4	C14—C13—H13B	111.5
C4—C3—H3	120.4	H13A—C13—H13B	109.3
C5—C4—C3	120.7 (3)	O3—C14—C13	105.4 (2)
C5—C4—H4	119.7	O3—C14—H14A	110.7
C3—C4—H4	119.7	C13—C14—H14A	110.7
C4—C5—C6	121.1 (3)	O3—C14—H14B	110.7
C4—C5—H5	119.4	C13—C14—H14B	110.7
C6—C5—H5	119.4	H14A—C14—H14B	108.8
C1—C6—C5	118.3 (2)	C20—C15—C16	118.1 (2)
C1—C6—N2	124.5 (2)	C20—C15—C7	122.7 (2)
C5—C6—N2	117.2 (2)	C16—C15—C7	119.2 (2)
N2—C7—C15	118.8 (2)	C17—C16—C15	119.9 (3)
N2—C7—C8	121.6 (2)	C17—C16—H16	120.0
C15—C7—C8	119.5 (2)	C15—C16—H16	120.0
C9—C8—C7	104.95 (17)	C18—C17—C16	120.7 (3)
C9—C8—H8A	110.8	C18—C17—H17	119.6
C7—C8—H8A	110.8	C16—C17—H17	119.6
C9—C8—H8B	110.8	C19—C18—C17	119.8 (3)
C7—C8—H8B	110.8	C19—C18—H18	120.1
H8A—C8—H8B	108.8	C17—C18—H18	120.1
O1—C9—N1	123.2 (2)	C18—C19—C20	120.0 (3)
O1—C9—C8	122.3 (2)	C18—C19—H19	120.0
N1—C9—C8	114.4 (2)	C20—C19—H19	120.0
N1—C10—C11	112.4 (2)	C15—C20—C19	121.4 (3)
N1—C10—H10A	109.1	C15—C20—H20	119.3
C11—C10—H10A	109.1	C19—C20—H20	119.3
N1—C10—H10B	109.1		

C9—N1—C1—C2	−132.7 (2)	C9—N1—C10—C11	−108.0 (2)
C10—N1—C1—C2	52.3 (3)	C1—N1—C10—C11	67.3 (3)
C9—N1—C1—C6	49.8 (3)	C12—N3—C11—C10	−137.1 (2)
C10—N1—C1—C6	−125.2 (2)	C13—N3—C11—C10	66.2 (3)
C6—C1—C2—C3	0.7 (4)	N1—C10—C11—N3	57.5 (3)
N1—C1—C2—C3	−176.8 (3)	C14—O3—C12—O2	176.3 (3)
C1—C2—C3—C4	0.7 (5)	C14—O3—C12—N3	−4.3 (3)
C2—C3—C4—C5	−0.6 (4)	C11—N3—C12—O2	12.8 (4)
C3—C4—C5—C6	−1.0 (4)	C13—N3—C12—O2	172.0 (3)
C2—C1—C6—C5	−2.2 (3)	C11—N3—C12—O3	−166.5 (2)
N1—C1—C6—C5	175.2 (2)	C13—N3—C12—O3	−7.3 (3)
C2—C1—C6—N2	−179.1 (2)	C12—N3—C13—C14	14.8 (3)
N1—C1—C6—N2	−1.6 (3)	C11—N3—C13—C14	173.5 (2)

C4—C5—C6—C1	2.4 (4)	C12—O3—C14—C13	13.4 (3)
C4—C5—C6—N2	179.5 (2)	N3—C13—C14—O3	−16.4 (3)
C7—N2—C6—C1	−42.9 (3)	N2—C7—C15—C20	161.8 (2)
C7—N2—C6—C5	140.2 (2)	C8—C7—C15—C20	−22.5 (3)
C6—N2—C7—C15	173.44 (19)	N2—C7—C15—C16	−19.4 (3)
C6—N2—C7—C8	−2.2 (3)	C8—C7—C15—C16	156.3 (2)
N2—C7—C8—C9	76.5 (3)	C20—C15—C16—C17	0.4 (4)
C15—C7—C8—C9	−99.1 (2)	C7—C15—C16—C17	−178.4 (2)
C1—N1—C9—O1	178.2 (2)	C15—C16—C17—C18	0.2 (4)
C10—N1—C9—O1	−6.8 (3)	C16—C17—C18—C19	−1.3 (4)
C1—N1—C9—C8	−5.5 (3)	C17—C18—C19—C20	1.8 (5)
C10—N1—C9—C8	169.45 (19)	C16—C15—C20—C19	0.1 (4)
C7—C8—C9—O1	107.4 (3)	C7—C15—C20—C19	178.9 (3)
C7—C8—C9—N1	−68.8 (2)	C18—C19—C20—C15	−1.2 (5)

Fig. 1


C10
C11
O1
N3
C2
C3
C1
N1
C13
C9
O2
C14
C12
C8
O3
C4
C6
C5
N2
C7
C15
C20
C19
C16
C18
C17