Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71528 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1033]

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Structure of OPC-21268

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(*Received* 21 *September* 1992; *accepted* 9 *July* 1993)

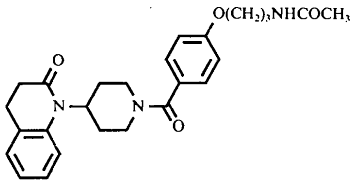
Abstract

The molecule of OPC-21268, *N*-(4-{[4-(2-oxo-1,2,3,4-tetrahydroquinolin-1-yl)piperidino]carbonyl}phenoxypropyl)acetamide, $C_{26}H_{31}N_3O_4$, has an extended conformation through the 3,4-dihydro-2(1*H*)-quinolone, piperidine, the phenyl rings and the acetamide side chain. The structure is stabilized in the crystal by an N—H···O intermolecular hydrogen bond [2.883 (4) Å].

Comment

OPC-21268 (1), synthesized by Ogawa *et al.* (1989), has been identified as an orally effective non-peptide vasopressin-V_1-receptor antagonist (Yamamura *et al.*, 1991) and is a potentially useful vasodilator. The study of its conformation might be useful in developing pharmacophores for the vasopressin-V_1-receptor.

We have determined the crystal structure of carteolol, a β-adrenergic blocking drug, which has a 3,4-dihydro-2(1*H*)-quinolone ring. The shortest C—C single bond of this ring was found to be 1.40 Å and the ring was planar (Kido, Nakagawa, Fujiwara & Tomita, 1981). It was considered that this abnormal structure was caused by either disordering or high thermal motion. We are interested in the ring structure of OPC-21268 as it is similar to that of carteolol.



(1)

The molecule has an extended conformation; distances between the N atoms are N(1)···N(2) 4.262 (4) and N(2)···N(3) 11.208 (5) Å. The piperidine ring has a perfect chair form with a *syn* conformation, χ[C(2)—N(1)—C(11)—C(12)] = 66.2 (4)°, against 3,4-dihydro-2(1*H*)-quinolone. There is an intermolecular hydrogen bond between NH of the terminal amide group and C=O of the benzoyl group in the middle of the molecule. This hydrogen bond may reduce the flexibility of the long molecule. The 3,4-dihydro-2(1*H*)-quinolone ring is clearly non-planar and no abnormal bond distances or angles were found.

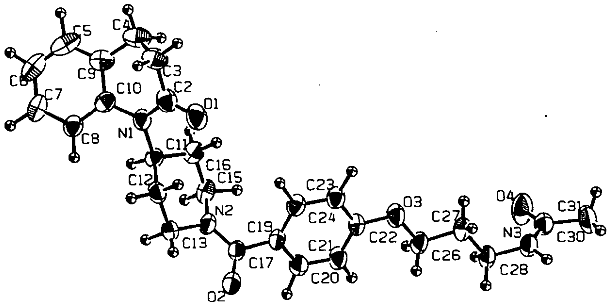


Fig. 1. View of OPC-21268 projected along the *a* axis showing the labeling of the non-H atoms. Thermal ellipsoids are shown at the 50% probability level; H atoms are drawn as small spheres of arbitrary radii.

Experimental

Crystal data

$C_{26}H_{31}N_3O_4$
$M_r = 449.55$
Monoclinic
$P2_1/c$
$a = 16.768$ (6) Å
$b = 8.958$ (3) Å
$c = 17.243$ (4) Å
$\beta = 113.50$ (2)°
$V = 2375$ (1) Å^3
$Z = 4$
$D_x = 1.257$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 5$–6°
$\mu = 0.080$ mm^{-1}
Ambient temperature
Rod-like
0.3 × 0.3 × 0.2 mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans [width (1.26 + 0.30tanθ)° in ω, speed 32° min^{-1}]
Absorption correction: none
3486 measured reflections
3354 independent reflections
1821 observed reflections [$I > 3\sigma(I)$]

$R_{\text{int}} = 0.038$
$\theta_{\max} = 22.5°$
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 10$
$l = -19 \rightarrow 19$
3 standard reflections monitored every 150 reflections intensity variation: none

Refinement

Refinement on F
$R = 0.043$
$wR = 0.049$
$S = 1.39$
1821 reflections
298 parameters
H atoms refined isotropically
$w = 4F_o^2/\sigma^2(F_o)^2$

$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection and cell refinement were carried out using Rigaku *MSC/AFC* software (Rigaku Corporation, 1988) and the structure was solved using *MITHRIL* (Gilmore, 1984). All calculations, including data reduction, were performed with the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985). Refinement was carried out using the full-matrix least-squares method. The positions of the H atoms were determined from the residual peaks in the final difference syntheses.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0.9113 (2)	0.3714 (3)	0.5217 (2)	5.3 (1)
O(2)	0.6714 (2)	−0.1466 (3)	0.3776 (1)	4.8 (1)
O(3)	0.4979 (1)	0.2508 (3)	0.0549 (1)	4.5 (1)
O(4)	0.2134 (2)	0.4542 (4)	−0.2187 (2)	7.0 (2)
N(1)	0.8656 (2)	0.3699 (4)	0.6291 (2)	3.8 (1)
N(2)	0.6915 (2)	0.0702 (3)	0.4488 (2)	3.6 (1)
N(3)	0.2941 (2)	0.2717 (4)	−0.2398 (2)	4.1 (1)
C(2)	0.9159 (2)	0.4253 (5)	0.5889 (3)	4.3 (2)
C(3)	0.9727 (3)	0.5552 (5)	0.6302 (3)	5.0 (2)
C(4)	0.9300 (3)	0.6570 (5)	0.6721 (3)	5.6 (2)
C(5)	0.9246 (3)	0.6141 (6)	0.8168 (3)	5.8 (2)
C(6)	0.9093 (3)	0.5227 (7)	0.8731 (3)	6.0 (3)
C(7)	0.8798 (3)	0.3815 (6)	0.8505 (3)	4.9 (2)
C(8)	0.8637 (2)	0.3288 (5)	0.7697 (2)	3.8 (2)
C(9)	0.9103 (2)	0.5664 (5)	0.7355 (3)	4.4 (2)
C(10)	0.8793 (2)	0.4219 (4)	0.7126 (2)	3.6 (2)
C(11)	0.7962 (2)	0.2592 (4)	0.5868 (2)	3.4 (2)
C(12)	0.8295 (3)	0.1087 (5)	0.5718 (3)	3.9 (2)
C(13)	0.7538 (3)	0.0031 (5)	0.5276 (2)	4.2 (2)
C(15)	0.6565 (2)	0.2132 (5)	0.4615 (2)	3.9 (2)
C(16)	0.7291 (3)	0.3238 (5)	0.5044 (2)	4.1 (2)
C(17)	0.6588 (2)	−0.0105 (5)	0.3777 (2)	3.5 (2)
C(19)	0.6105 (2)	0.0682 (4)	0.2959 (2)	3.3 (2)
C(20)	0.5327 (3)	0.0117 (5)	0.2384 (2)	4.2 (2)
C(21)	0.4918 (3)	0.0713 (5)	0.1580 (2)	4.3 (2)
C(22)	0.5304 (2)	0.1876 (4)	0.1334 (2)	3.4 (2)
C(23)	0.6078 (2)	0.2469 (5)	0.1901 (2)	3.8 (2)
C(24)	0.6466 (2)	0.1878 (5)	0.2710 (2)	3.7 (2)
C(26)	0.4199 (3)	0.1894 (5)	−0.0074 (2)	4.0 (2)
C(27)	0.4007 (3)	0.2643 (5)	−0.0910 (2)	3.8 (2)
C(28)	0.3176 (3)	0.2033 (5)	−0.1574 (2)	4.1 (2)
C(30)	0.2406 (3)	0.3879 (5)	−0.2656 (2)	4.3 (2)
C(31)	0.2141 (5)	0.4317 (8)	−0.3560 (3)	6.2 (3)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(2)	1.229 (5)	C(6)—C(7)	1.357 (7)
O(2)—C(17)	1.237 (5)	C(7)—C(8)	1.392 (6)
O(3)—C(22)	1.365 (4)	C(8)—C(10)	1.392 (5)
O(3)—C(26)	1.431 (5)	C(9)—C(10)	1.393 (6)
O(4)—C(30)	1.227 (5)	C(11)—C(12)	1.520 (6)
N(1)—C(2)	1.382 (5)	C(11)—C(16)	1.532 (6)
N(1)—C(10)	1.441 (5)	C(12)—C(13)	1.523 (6)
N(1)—C(11)	1.480 (5)	C(15)—C(16)	1.514 (6)
N(2)—C(13)	1.472 (5)	C(17)—C(19)	1.494 (5)
N(2)—C(15)	1.461 (5)	C(19)—C(20)	1.382 (6)
N(2)—C(17)	1.338 (5)	C(19)—C(24)	1.381 (6)
N(3)—C(28)	1.450 (5)	C(20)—C(21)	1.386 (6)
N(3)—C(30)	1.330 (5)	C(21)—C(22)	1.379 (6)
C(2)—C(3)	1.493 (6)	C(22)—C(23)	1.381 (5)
C(3)—C(4)	1.510 (7)	C(23)—C(24)	1.388 (5)
C(4)—C(9)	1.500 (6)	C(26)—C(27)	1.504 (6)
C(5)—C(6)	1.370 (7)	C(27)—C(28)	1.509 (6)
C(5)—C(9)	1.391 (6)	C(30)—C(31)	1.494 (6)
C(22)—O(3)—C(26)	118.0 (3)	C(12)—C(11)—C(16)	111.2 (4)
C(2)—N(1)—C(10)	120.0 (4)	C(11)—C(12)—C(13)	110.3 (4)
C(2)—N(1)—C(11)	120.7 (3)	N(2)—C(13)—C(12)	110.6 (4)
C(10)—N(1)—C(11)	119.4 (3)	N(2)—C(15)—C(16)	110.8 (4)
C(13)—N(2)—C(15)	113.5 (4)	C(11)—C(16)—C(15)	110.8 (4)
C(13)—N(2)—C(17)	120.4 (4)	O(2)—C(17)—N(2)	121.7 (4)
C(15)—N(2)—C(17)	124.7 (4)	O(2)—C(17)—C(19)	119.5 (4)
C(28)—N(3)—C(30)	122.8 (4)	N(2)—C(17)—C(19)	118.7 (4)
O(1)—C(2)—N(1)	120.8 (4)	C(17)—C(19)—C(20)	120.4 (4)
O(1)—C(2)—C(3)	123.0 (4)	C(17)—C(19)—C(24)	121.4 (4)
N(1)—C(2)—C(3)	116.2 (4)	C(20)—C(19)—C(24)	117.8 (4)
C(2)—C(3)—C(4)	111.4 (4)	C(19)—C(20)—C(21)	121.7 (4)
C(3)—C(4)—C(9)	107.9 (4)	C(20)—C(21)—C(22)	119.5 (4)
C(6)—C(5)—C(9)	121.6 (5)	O(3)—C(22)—C(21)	124.6 (4)
C(5)—C(6)—C(7)	120.2 (5)	O(3)—C(22)—C(23)	115.7 (4)
C(6)—C(7)—C(8)	120.4 (5)	C(21)—C(22)—C(23)	119.8 (4)
C(7)—C(8)—C(10)	119.3 (5)	C(22)—C(23)—C(24)	119.7 (4)
C(4)—C(9)—C(5)	124.7 (5)	C(19)—C(24)—C(23)	121.4 (4)
C(4)—C(9)—C(10)	117.5 (4)	O(3)—C(26)—C(27)	109.3 (4)
C(5)—C(9)—C(10)	117.8 (5)	C(26)—C(27)—C(28)	110.3 (4)
N(1)—C(10)—C(8)	120.8 (4)	N(3)—C(28)—C(27)	113.5 (4)
N(1)—C(10)—C(9)	118.5 (4)	O(4)—C(30)—N(3)	122.5 (4)
C(8)—C(10)—C(9)	120.7 (4)	O(4)—C(30)—C(31)	121.8 (5)
N(1)—C(11)—C(12)	114.1 (3)	N(3)—C(30)—C(31)	115.7 (5)
N(1)—C(11)—C(16)	110.3 (3)		

D—H···A	D···A
N(3)—H(3)···O(2$^{\text{i}}$)	2.883 (4)

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

Lists of structure factors, anisotropic displacement parameters, torsion angles and H-atom coordinates and geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71487 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1017]

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Acta Cryst. (1994). C**50**, 304–306

1,6-Diselenacyclodecane-3,4,8,9-tetraone Tetrakis(*O*-methyloxime)

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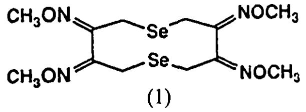
(*Received* 1 *February* 1993; *accepted* 18 *June* 1993)

Abstract

The diselenacyclodecane ring in the title compound, $C_{12}H_{20}N_4O_4Se_2$, adopts a twist–twist–boat conformation with regular *trans* positions of the oxime groups. In this conformation, the intramolecular spatial interaction of the oxime fragments decreases to a minimum. This molecule is an *E,E,E,E* isomer with regard to the configuration of the oxime groups.

Comment

As part of our research on oxime-based macrocyclization (Shatzmiller, Menashe, Shalom & Bahar, 1991), the structure determination of 1,6-diselenacyclodecane-3,4,8,9-tetraone tetrakis(*O*-methyloxime) (1) was undertaken in order to establish the configuration of one of three products obtained from the reaction of 1,4-dibromobutan-2,3-dione bis(*O*-methyloxime) with disodium selenide.



(1)

Fig. 1 illustrates the top and side views of the conformation of (1) in its molecular crystal. In contrast to diselena[3.3]orthocyclophane, which in the solid state has a chair conformation (Okajima, Wang & Fukazawa, 1989), the diselenacyclodecane ring of (1) adopts a twist–twist–boat conformation. In this conformation, the intramolecular spatial interaction of the oxime moieties decreases to a minimum, but at the same time the possibility for conjugation in the —N=C—C=N— moiety is preserved. The C4—C3—N11—O12, C3—C4—N14—O15, C9—C8—N17—O18 and C8—C9—N20—O21 torsion angles are 179.4 (4), 180.0 (4), 180.0 (4) and −178.7 (4)°, respectively. These values, and Fig. 1, indicate that (1) is an *E,E,E,E* isomer with regard to the configuration of the oxime groups.

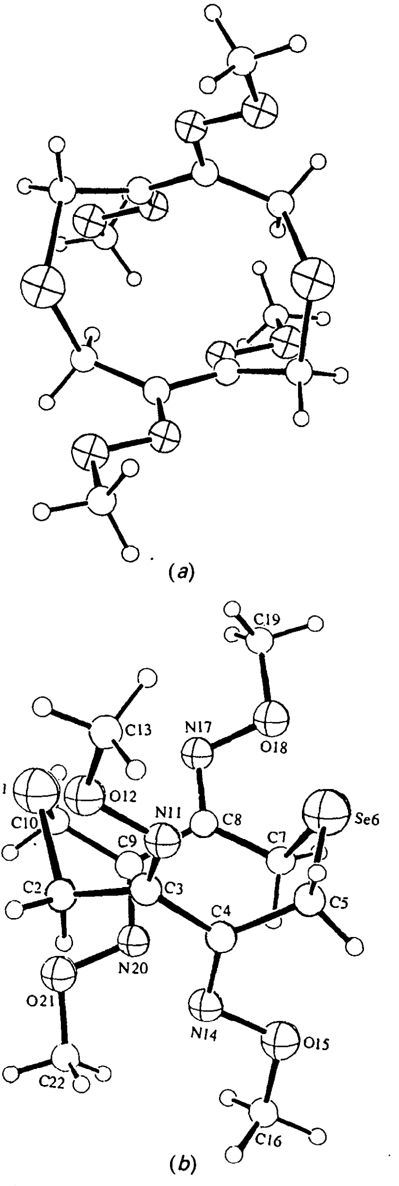


Fig. 1. Molecular conformation of the title compound: (*a*) top and (*b*) side views, showing the atom labelling. Atoms are of arbitrary size, the N, O and Se atoms being marked by crossed circles.