C17	0.2946 (4)	0.2609 (3)	0.0810 (7)	0.0361
C18	0.3304 (3)	0.2976 (3)	0.0974 (7)	0.0360
C19	0.3783 (3)	0.2640 (3)	−0.0552 (8)	0.0353
C20	0.3427 (3)	0.2298 (3)	−0.0733 (8)	0.0327
C21	0.3563 (5)	0.3822 (4)	0.1536 (10)	0.0787
C22	0.2021 (3)	0.2387 (4)	0.0881 (9)	0.0502
C23	0.2839 (4)	0.1696 (4)	0.0275 (8)	0.0427
C24	0.3038 (3)	0.1217 (3)	−0.0180 (8)	0.0365
C25	0.2710 (4)	0.0841 (4)	−0.0275 (9)	0.0545
C26	0.2872 (5)	0.0406 (4)	−0.0729 (12)	0.0664
C27	0.3341 (6)	0.0335 (4)	−0.1059 (10)	0.0672
C28	0.3662 (4)	0.0710 (4)	−0.0942 (9)	0.0533
C29	0.3507 (3)	0.1149 (4)	−0.0499 (8)	0.0360
C30	0.2180 (4)	0.0891 (4)	0.0044 (11)	0.0813

Table 4. *Selected geometric parameters* (Å, °) *for isomer* (II)

C9—C12	1.57 (1)	C10—C18	1.52 (1)
C9—C17	1.57 (1)	C10—C19	1.50 (2)
C9—C20	1.55 (1)	C11—C12	1.40 (1)
C9—C23	1.55 (1)	C17—C18	1.44 (1)
C10—C11	1.52 (2)	C19—C20	1.39 (1)
C12—C9—C17	104.9 (7)	C18—C10—C19	106.7 (8)
C12—C9—C20	108.3 (7)	C10—C11—C12	112.0 (8)
C12—C9—C23	109.9 (7)	C9—C12—C11	113.8 (8)
C17—C9—C20	101.8 (7)	C9—C17—C18	110.6 (8)
C17—C9—C23	115.0 (7)	C10—C18—C17	113.7 (8)
C20—C9—C23	116.1 (7)	C10—C19—C20	114.0 (8)
C11—C10—C18	105.8 (8)	C9—C20—C19	112.7 (8)
C11—C10—C19	107.6 (8)		

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71554 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1064]

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5,5-Diphenyl-1*H*,2*H*,5*H*-imidazo[1,2-*a*]imidazole-3,6-dione

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Abstract

The conformation of the title molecule, $C_{17}H_{13}N_3O_2$, is generally similar to the conformations of analogous compounds. Both five-membered rings possess envelope conformations. The dihedral angle between the mean planes of these two rings equals 5.33 (8)°. The molecules in the crystal are joined by an intermolecular hydrogen bond N1—H4···O1(x, $\frac{1}{2}+y$, z) of 2.869 (3) Å.

Comment

Hydantoins are classified as compounds that influence the central nervous system (CNS) (Vida, 1977). The crystal and molecular structures of pharmacologically active hydantoins have been the subject of many papers (Heeg, Subramanian & Dryhurst, 1986; Camerman, Mastropaolo & Camerman, 1983; Uno

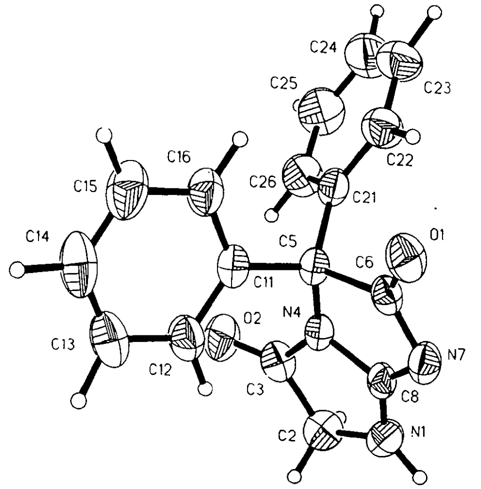

Fig. 1. Molecular structure and atomic numbering scheme for the title compound.

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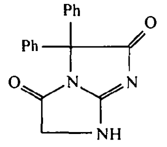
& Shimizu, 1980; Foresti, Sabatino, Riva di Sanseverino, Fusco, Tosi & Tonani, 1988; Gallueci, Mathur & Schechter, 1992). Searching for new compounds with the potential to influence the CNS we have examined annelated 5,5-diphenyl-2-thiohydantoin (DPTH) (Kieć-Kononowicz, Zatorski & Karolak-Wojciechowska, 1989) and 5-substituted benzylidene-2-thiohydantoin derivatives (Kieć-Kononowicz & Karolak-Wojciechowska, 1992). The DPTH derivatives exhibit sedative properties while the analogous derivatives of 5-substituted benzylidene-2-thiohydantoin have analgetic, anxiolitic, antidepressant and anticonvulsant properties (Kolasa, Kleinrok, Pietrusiewicz, Czechowski, Kieć-Kononowicz & Zejc, 1989). The aim of this structure determination is to obtain three-dimensional data for the pharmacologically tested (inactive) derivative (I),



(I)

i.e. a hydantoin derivative with oxygen isosterically substituted by nitrogen (Lambert, Evrard & Durant, 1978). These data will be used for the calculation and comparison of the molecular electrostatic potential distribution round the active and inactive molecules. A similar procedure for phenylsuccinimides offered the possibility of predicting active compounds based on the calculations alone (Kwiatkowski & Karolak-Wojciechowska, 1993).

Experimental

Crystal data

$C_{17}H_{13}N_3O_2$
$M_r = 291.31$
Orthorhombic
$P2_12_12_1$
$a = 15.829$ (4) Å
$b = 10.290$ (3) Å
$c = 8.529$ (5) Å
$V = 1389.21$ Å^3
$Z = 4$
$D_x = 1.393$ Mg m^{-3}
$D_m = 1.389$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10$-$60°$
$\mu = 0.84$ mm^{-1}
$T = 293$ K
Needles
0.4 × 0.2 × 0.2 mm
Colourless
Crystal source: crystallized from $CHCl_3$

Data collection

Kuma KM-4 diffractometer
ω-2θ scans
Absorption correction: none
1415 measured reflections
1415 independent reflections
1221 observed reflections [$F > 4\sigma(F)$]

$\theta_{max} = 55°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 8$
2 standard reflections monitored every 50 reflections intensity variation: 2%

Refinement

Refinement on F
$R = 0.032$
$wR = 0.036$
$S = 1.17$
1221 reflections
239 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F) + 0.0117F^2]$
$(\Delta/\sigma)_{max} = 0.01$

$\Delta\rho_{max} = 0.122$ e Å^{-3}
$\Delta\rho_{min} = -0.121$ e Å^{-3}
Extinction correction: empirical
Extinction coefficient: 0.01137
Atomic scattering factors from *SHELXTL/PC* (Sheldrick, 1990)

The structure was solved by direct methods using *SHELXTL/PC* (Sheldrick, 1990). All calculations were performed using the *SHELXTL/PC* system.

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.5227 (1)	0.3065 (2)	0.1175 (3)	0.0480 (4)
O2	0.2294 (1)	0.5428 (2)	−0.1117 (3)	0.0570 (4)
N1	0.3639 (1)	0.6771 (2)	0.1717 (3)	0.045 (1)
C2	0.2882 (2)	0.6960 (3)	0.0790 (4)	0.051 (1)
C3	0.2802 (1)	0.5700 (3)	−0.0126 (3)	0.0430 (4)
N4	0.3456 (1)	0.4934 (2)	0.0426 (2)	0.0350 (4)
C5	0.3828 (1)	0.3653 (2)	0.0142 (3)	0.0340 (4)
C6	0.4658 (1)	0.3863 (3)	0.1146 (3)	0.0360 (4)
N7	0.4652 (1)	0.5026 (2)	0.1914 (3)	0.0410 (4)
C8	0.3955 (1)	0.5613 (2)	0.1455 (3)	0.0350 (4)
C11	0.4069 (1)	0.3457 (3)	−0.1572 (3)	0.0370 (4)
C12	0.4185 (2)	0.4516 (3)	−0.2563 (4)	0.049 (1)
C13	0.4438 (2)	0.4324 (4)	−0.4113 (4)	0.060 (1)
C14	0.4557 (2)	0.3075 (4)	−0.4661 (4)	0.066 (1)
C15	0.4440 (2)	0.2031 (4)	−0.3697 (4)	0.065 (1)
C16	0.4204 (2)	0.2215 (3)	−0.2138 (4)	0.049 (1)
C21	0.3280 (1)	0.2567 (3)	0.0831 (3)	0.0380 (4)
C22	0.3632 (2)	0.1538 (3)	0.1656 (4)	0.052 (1)
C23	0.3117 (2)	0.0592 (3)	0.2314 (5)	0.068 (1)
C24	0.2258 (2)	0.0653 (4)	0.2144 (5)	0.076 (1)
C25	0.1902 (2)	0.1661 (4)	0.1303 (5)	0.070 (1)
C26	0.2407 (2)	0.2611 (3)	0.0627 (4)	0.054 (1)

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

O1—C6	1.219 (3)	N4—C8	1.372 (3)
O2—C3	1.200 (3)	C5—C6	1.583 (3)
N1—C2	1.449 (4)	C5—C11	1.524 (4)
N1—C8	1.311 (3)	C5—C21	1.532 (3)
C2—C3	1.519 (4)	C6—N7	1.364 (4)
C3—N4	1.384 (3)	N7—C8	1.317 (3)
N4—C5	1.464 (3)	C(Ph)—C(Ph) av.	1.385 (5)
C2—N1—C8	110.1 (2)	O1—C6—C5	122.2 (2)
N1—C2—C3	103.6 (2)	O1—C6—N7	125.9 (2)
O2—C3—C2	128.1 (2)	C5—C6—N7	112.0 (2)
O2—C3—N4	127.5 (2)	C6—N7—C8	105.4 (2)
C2—C3—N4	104.4 (2)	N1—C8—N4	110.6 (2)
C3—N4—C5	139.3 (2)	N1—C8—N7	133.3 (2)
C3—N4—C8	111.0 (2)	N4—C8—N7	116.0 (2)
C5—N4—C8	109.4 (2)	C5—C11—C12	120.8 (2)
N4—C5—C6	97.0 (2)	C5—C11—C16	119.9 (2)
N4—C5—C11	112.2 (2)	C5—C21—C22	121.6 (2)
N4—C5—C21	111.5 (2)	C5—C21—C26	119.3 (2)
C6—C5—C11	109.2 (2)	C22—C21—C26	119.1 (3)
C6—C5—C21	111.2 (2)	C(Ph)—C(Ph)—C(Ph) av.	120.0 (3)
C11—C5—C21	114.4 (2)		

The crystallographic investigations were supported by grant No. 303029101 from the Polish State Committee for Scientific Research.

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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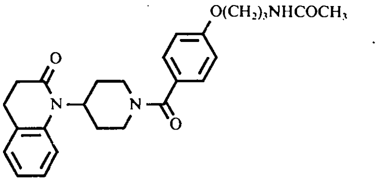
Abstract

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.

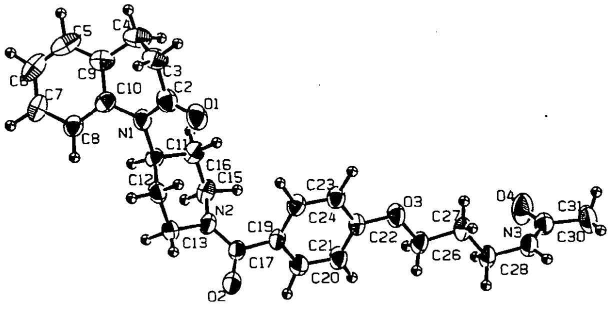


Fig. 1. View of OPC-21268 projected along the *a* axis showing the labeling of the non-H atoms. Thermal ellipsoids are shown at the 50% probability level; H atoms are drawn as small spheres of arbitrary radii.

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