Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71337 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1036]

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Structure of 2-(2-Hydroxyethyl)-1-*p*-tolyl-4,5,6,7-tetrahydro-3(1*H*)-isoindolone

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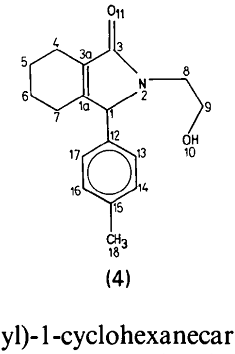
Abstract

The flexible cyclohexene ring of the isoindole moiety, as indicated by the positional disorder of the C5 and C6 atoms, assumes two kinds of half-chair conformation, $^4H_5$ or $^5H_4$, at an equilibrium ratio of 68:32. The planar pyrrole ring forms dihedral angles of 76.45 (3) and 86.21 (9)° with the best planes through the 1-*p*-tolyl and 2-ethyl moieties, respectively. Both moieties occupy a *cis* position with respect to the pyrrole ring [N2—C1—C12—C13 = 98.9 (2), C1—N2—C8—C9 = 82.0 (2)°].

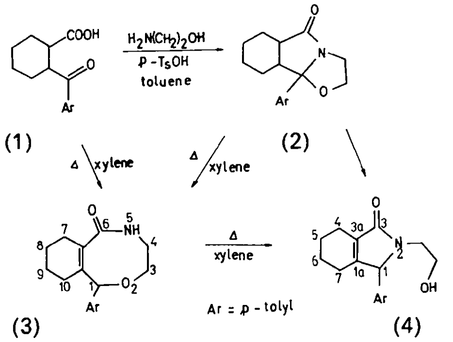
Comment

The title compound (4) was synthesized with pharmacological aims (Stájer *et al.*, 1992, unpublished results), because it is known that similar structures, including different isoindoles condensed with heterocycles, have a potentially anorexigenic effect (Orzalesi *et al.*, 1978).



(4)

cis-2-(*p*-Toluoyl)-1-cyclohexanecarboxylic acid (1) was refluxed with ethanolamine in toluene, in the presence of *p*-toluenesulfonic acid as catalyst and 9b-*p*-tolyl-2,3,5a,6,7,8,9,9a-octahydrooxazolo[2,3-*a*]isoindol-5(9b*H*)-one (2) was obtained. Under similar conditions in xylene, (1) afforded 1-*p*-tolyl-1*H*-3,4,7,8,9,10-hexahydro-2,5-benzoxazocin-6(5*H*)-one (3) which could also be prepared from (2) by heating in xylene. (2) may undergo transformation to give (3) or 2-(2-hydroxyethyl)-1-*p*-tolyl-4,5,6,7-tetrahydroisoindol-3(1*H*)-one (4). The isomeric structures of (3) and (4) cannot be differentiated by means of NMR because the signals are merged and the splits are similar. The X-ray data prove structure (4), which may be formed from (2) by simple hydrolysis, but the ring transformation to (3) and subsequent hydrolysis can also be supposed.



The structure was refined for the two most probable positions of atoms C5 and C6, given in Table 1, which indicates the conformational disorder (Fig. 1). The puckering parameters (Cremer & Pople, 1975) of cyclohexene ring conformations C [C1a—C3a—C4—C5—C6—C7: $Q = 0.484$ (5) Å, $\varphi = 32.4$ (8), $\theta = 130.0$ (6)°] and C' [C1a—C3a—C4—C5′—C6′—C7: $Q = 0.46$ (1) Å, $\varphi = 211(2)$°, $\theta = 49$ (2)°] show that it adopts two kinds of half-chair form, $^4H_5$ and $^5H_4$, respectively (Boeyens, 1978). The increased motion of the atomic displacements of the peripheral atoms

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and the low melting point of the crystal (m.p. 383–384 K) are in accordance with the obtained conformational equilibrium. [Note also the existence of the double bond C1*a*=C3*a* = 1.333 (2) Å, since the double bond is prerequisite to a flexible cyclohexene ring.] Molecular-mechanical calculations performed by the *MMX* program (Burkert & Allinger, 1982; Allinger, Schäfer, Siam, Klinkowski & van Alsenoy, 1985) show that both initial models of the molecule with ring conformations *C* and *C′* lead to the same energy. As shown in the literature (Argay, Kálmán, Párkányi, Ribár & Djarmati, 1991) more than two energetically similar conformers of flexible molecules frequently exist together in the crystalline state. The present work reports on an equilibrium of two conformers *C* and *C′* retained in the solid state with a ratio of 68:32.

Apart from C5 and C6, the condensed isoindole moiety is planar. The best planes through the cyclohexene and pyrrole rings form an angle of 0.73 (5)°. The significantly shortened bond length N2—C3 = 1.349 (2) Å around N2 can be explained by the influence of the delocalized π-electron cloud along part of pyrrole ring (C1*a*—C3*a*—C3—N2). Relevant torsion angles (C1—N2—C8—C9, N2—C1—C12—C13) (Table 2) indicate that the 2-ethyl and 1-*p*-tolyl groups form angles of −82.0 (2) and 98.9 (2)°, respectively, relative to the isoindole moiety, since the terminal oxygen O10 is synclinal with N2 [N2—C8—C9—O10 = 61.4 (2)°]. The value of torsion angle N2—C1—C12—C13 [98.9 (2)°] differs to a certain degree from the value determined from the energy-minimized free-molecule structure (124.5°) calculated using *MMX* (Allinger *et al.*, 1985; Burkert & Allinger, 1982). This difference can be explained by the effect of relatively strong intramolecular van der Waals repulsion between the H13 and H10 atoms [2.70 (3) Å in the crystal and 3.55 Å in the free molecule] which is probably the effect of molecular packing in the crystal due to the strong intermolecular O10—H10···O11′ hydrogen bond. Molecules bonded by the intermolecular hydrogen bond (Fig. 2) [O10···O11 = 2.779 (2), H10···O11 = 1.680 (25) Å, O10—H10···O11 = 173.4 (23)° (x, $\frac{1}{2}-y$, $\frac{1}{2}+z$)] form layers parallel to the *bc* plane.

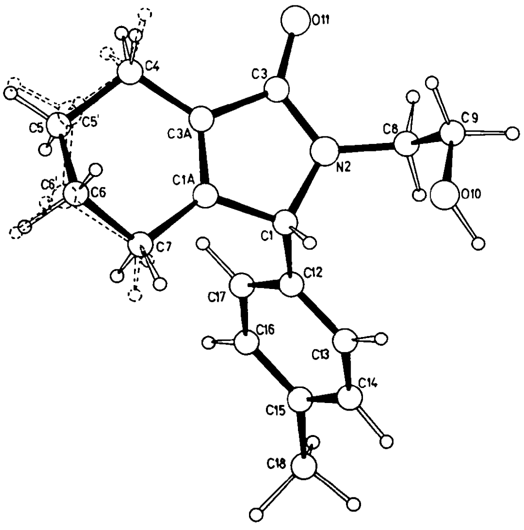

Fig. 1. Perspective view of the molecule showing the atomic numbering. The H atoms are shown but not labelled.

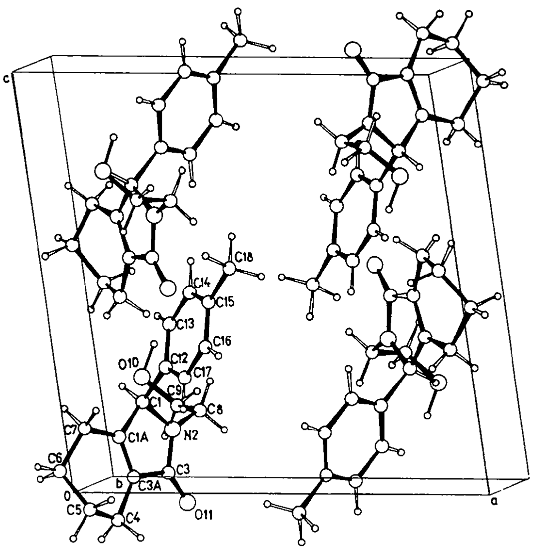

Fig. 2. Packing diagram of the unit cell projected along the *b* axis.

Experimental

Crystal data

$C_{17}H_{21}NO_2$
M_r = 271.65
Monoclinic
$P2_1/c$
a = 13.146 (1) Å
b = 8.917 (1) Å
c = 12.829 (1) Å
β = 98.89 (1)°
V = 1485.8 (2) Å^3
Z = 4

D_x = 1.214 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 11.45–45.27°
μ = 0.59 mm^{-1}
T = 293 K
Prism
0.3 × 0.25 × 0.1 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω-θ scans
Absorption correction: by integration from crystal shape
$T_{\min}$ = 0.9564, $T_{\max}$ = 0.9984
3222 measured reflections
3011 independent reflections

2480 observed reflections [$F > 4\sigma(F)$]
$\theta_{\max}$ = 74°
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity variation: 0.05%

Refinement

Refinement on F
Final R = 0.044
wR = 0.046
S = 2.240
2595 [$F > 3\sigma(F)$] reflections
243 parameters
$w = 5.3187/[\sigma^2(F) + 0.000096F^2]$
$(\Delta/\sigma)_{\max}$ = 0.125 (H81, z coordinate)
$\Delta\rho_{\max}$ = 0.17 e Å^{-3}
$\Delta\rho_{\min}$ = −0.19 e Å^{-3}
Atomic scattering factors from *SHELX*76

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

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

	x	y	z	U_{eq}
C1	0.1987 (1)	0.0049 (2)	1.2181 (1)	0.379 (3)
C1*a*	0.1413 (1)	−0.1009 (2)	1.1383 (1)	0.372 (3)
N2	0.2516 (1)	0.1020 (1)	1.1506 (1)	0.405 (3)
C3	0.2288 (1)	0.0636 (2)	1.0479 (1)	0.388 (3)
C3*a*	0.1595 (1)	−0.0675 (2)	1.0416 (1)	0.367 (3)
C4	0.1151 (1)	−0.1482 (2)	0.9431 (1)	0.449 (3)
C5	0.0663 (4)	−0.2982 (5)	0.9728 (3)	0.519 (8)
C5′	0.0284 (8)	−0.2447 (11)	0.9627 (6)	0.546 (17)
C6	0.0034 (3)	−0.2770 (5)	1.0623 (3)	0.497 (7)
C6′	0.0524 (10)	−0.3266 (9)	1.0662 (5)	0.592 (20)
C7	0.0725 (1)	−0.2247 (2)	1.1631 (1)	0.462 (4)
C8	0.3093 (1)	0.2345 (2)	1.1890 (1)	0.467 (4)
C9	0.2423 (2)	0.3712 (2)	1.1952 (1)	0.560 (4)
O10	0.1700 (1)	0.3542 (2)	1.2645 (1)	0.682 (4)
O11	0.2607 (1)	0.1299 (1)	0.9747 (1)	0.531 (3)
C12	0.2736 (1)	−0.0702 (2)	1.3039 (1)	0.382 (3)
C13	0.2905 (1)	−0.0130 (2)	1.4055 (1)	0.480 (4)
C14	0.3590 (1)	−0.0821 (2)	1.4835 (1)	0.544 (4)
C15	0.4120 (1)	−0.2106 (2)	1.4638 (1)	0.506 (4)
C16	0.3956 (1)	−0.2670 (2)	1.3619 (1)	0.540 (4)
C17	0.3279 (1)	−0.1980 (2)	1.2830 (1)	0.484 (4)
C18	0.4833 (2)	−0.2886 (3)	1.5507 (2)	0.758 (6)

Table 2. *Geometric parameters* (Å, °)

C1—C1*a*	1.506 (2)	C5′—C6′	1.505 (11)
C1—N2	1.473 (2)	C6—C7	1.534 (4)
C1—C12	1.515 (2)	C6′—C7	1.530 (7)
C1*a*—C3*a*	1.333 (2)	C8—C9	1.513 (3)
C1*a*—C7	1.492 (2)	C9—O10	1.406 (3)
N2—C3	1.349 (2)	C12—C13	1.385 (2)
N2—C8	1.449 (2)	C12—C17	1.393 (2)
C3—C3*a*	1.477 (2)	C13—C14	1.383 (2)
C3—O11	1.236 (2)	C14—C15	1.384 (2)
C3*a*—C4	1.493 (2)	C15—C16	1.386 (2)
C4—C5	1.556 (5)	C15—C18	1.511 (3)
C4—C5′	1.480 (10)	C16—C17	1.384 (2)
C5—C6	1.527 (6)		
C1*a*—C1—N2	101.6 (1)	C4—C5′—C6′	111.7 (7)
C1*a*—C1—C12	114.7 (1)	C5—C6—C7	110.7 (3)
N2—C1—C12	112.0 (1)	C5′—C6—C7	121.1 (5)
C1—C1*a*—C3*a*	110.5 (1)	C5—C6′—C7	128.2 (7)
C1—C1*a*—C7	125.1 (1)	C5′—C6′—C7	114.5 (7)
C3*a*—C1*a*—C7	124.4 (1)	C1*a*—C7—C6	110.2 (2)
C1—N2—C3	111.8 (1)	C1*a*—C7—C6′	107.5 (4)
C1—N2—C8	123.3 (1)	N2—C8—C9	113.4 (1)
C3—N2—C8	124.4 (1)	C8—C9—O10	113.7 (2)
N2—C3—C3*a*	107.3 (1)	C1—C12—C13	120.8 (1)
N2—C3—O11	124.7 (1)	C1—C12—C17	121.1 (1)
C3*a*—C3—O11	128.0 (1)	C13—C12—C17	118.0 (1)
C1*a*—C3*a*—C3	108.9 (1)	C12—C13—C14	120.6 (1)
C1*a*—C3*a*—C4	125.3 (1)	C13—C14—C15	121.8 (1)
C3—C3*a*—C4	125.8 (1)	C14—C15—C16	117.6 (1)
C3*a*—C4—C5	109.2 (2)	C14—C15—C18	121.2 (2)
C3*a*—C4—C5′	110.3 (4)	C15—C16—C17	121.1 (1)
C4—C5—C6	111.7 (3)	C12—C17—C16	120.9 (1)
C1—C1*a*—C3*a*—C4	−179.7 (1)	C1*a*—C1—C12—C17	34.4 (2)
C1—N2—C3—O11	177.8 (1)	C3—N2—C8—C9	89.1 (2)
C1—N2—C8—C9	−82.0 (2)	C3—C3*a*—C1*a*—C7	178.5 (1)
C1*a*—C1—N2—C8	173.1 (1)	C7—C1*a*—C1—C12	59.7 (2)
N2—C8—C9—O10	61.4 (2)	C8—N2—C1—C12	−64.0 (2)
N2—C1—C12—C13	98.9 (2)	C8—N2—C3—O11	5.8 (2)
C1*a*—C1—C12—C13	−146.0 (1)	C4—C3*a*—C3—O11	1.1 (3)

Data were corrected for Lorentz and polarization factors. The structure was solved by direct methods with *SHELXS*86 (Sheldrick, 1986). The program used to refine the structure was *SHELX*76 (Sheldrick, 1976). After the isotropic refinement, the difference Fourier map showed the positionally disordered atoms C5 and C6. Introducing four disordered heavy-atom positions C5, C5′, C6, C6′, the anisotropic refinement converged with final occupancy factors of 0.68 (1) for atoms C5 and C6, and 0.32 (1) for atoms C5′, C6′. The positions of H atoms were generated from assumed geometries and treated isotropically in the final stage of the refinement. However, the H atoms bonded to C4, C5, C6, C5′, C6′, C7 and the methyl H atoms bonded to C18 were taken to have fixed positions and only an overall isotropic temperature factor was refined up to U = 0.066 (2) and 0.140 (6) Å^2, respectively. Conformational calculations were performed using the program *RING* (Párkányi, 1979). *CSU* (Vicković, 1988) was used to prepare the material for publication. All calculations were carried out on a PC/AT computer.

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