

The torsion angles $\tau[\text{C}(2)\text{—N}(1)\text{—C}(24)\text{—C}(25)] + 88.3 (8)^\circ$, $\tau[\text{N}(1)\text{—C}(24)\text{—C}(25)\text{—O}(2)] + 63.5 (8)^\circ$ and $\tau[\text{C}(24)\text{—C}(25)\text{—O}(2)\text{—C}(26)] - 110.4 (8)^\circ$ show that the side chain assumes a folded conformation. While in the majority of cases, these angles are close to 180° , a few of alkyl-substituted esters have values around 120° (Dunitz & Schweizer, 1982).

The intermolecular contacts (Fig. 3) are van der Waals.

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Acta Cryst. (1986). C42, 1549–1551

6,6-Dimethyl-4-oxo-1,2-diphenyl-4,5,6,7-tetrahydroindole

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(Received 6 June 1985; accepted 30 June 1986)

Abstract. $\text{C}_{22}\text{H}_{21}\text{NO}$, $M_r = 315.410$, $P\bar{1}$, $a = 9.316 (2)$, $b = 9.742 (4)$, $c = 11.057 (2) \text{ \AA}$, $\alpha = 98.560 (14)$, $\beta = 65.690 (15)$, $\gamma = 102.020 (25)^\circ$, $V = 892.2 \text{ \AA}^3$, $Z = 2$, $D_x = 1.175 \text{ Mg m}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$, $\mu = 0.32 \text{ mm}^{-1}$, $F(000) = 336$, $T = 293 \text{ K}$, $R = 0.058$ for 1600 reflections. The presence of conjugation between the pyrrole N atom and the carbonyl O atom, which is believed to cause the lack of activity of the 4-keto function in unsubstituted tetrahydroindoles, is not removed by the substituents in the title compound. The cyclohexene ring assumes a distorted sofa conformation.

Introduction. A majority of 4-ketotetrahydroindoles, particularly the unsubstituted compounds, were found to be inactive, even though a few of them are good analgesic, antipsychotic and antianxiety agents (Remers, Roth & Weiss, 1971). The lack of activity was attributed to the conjugation between the pyrrole nitrogen and the carbonyl function (Weiss, Allen, Gibbs, Pidacks, Poletto & Remers, 1968). The title compound is one of a series of 4-ketotetrahydroindole

derivatives prepared with the intention of delocalizing the lone pair of electrons of the N atom into the aromatic nucleus and activating the 4-keto function (Ramadas, Ramana & Padmanabhan, 1978).

Experimental. Title compound prepared by condensing 5,5-dimethyl-2-phenacyl-1,3-cyclohexanedione with aniline in refluxing glacial acetic acid. Recrystallization (by slow evaporation) from ethanol gave plate-shaped crystals. Approximate dimensions of crystal: $0.55 \times 0.20 \times 0.60 \text{ mm}$. Enraf–Nonius CAD-4 automated diffractometer, graphite monochromator. Intensities measured with NaI(Tl) scintillation counter interfaced with PDP/8a microcomputer. Cell parameters refined by least-squares method on basis of $25 \ 2\theta$ values. $2 < 2\theta < 30^\circ$. No significant variation in intensities of 2 check reflections, monitored every 100 reflections. Lp correction. Absorption neglected. 3054 unique reflections measured, 1600 with $I > 3\sigma(I)$. $h = \pm 7$, $k = \pm 9$, $l = \pm 11$. Space group from intensity statistics. Structure solved by multiscan technique using *MULTAN80* (Main *et al.*, 1980). Refinement of structure by least-squares method, H-atom positions from difference Fourier maps at an intermediate stage

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Table 1. *Final fractional atomic coordinates* ($\times 10^4$)
with *e.s.d.'s* in parentheses

$$U_{eq} = \sum_{i=1}^3 U_{ii}/3 \quad (\times 10^3 \text{ \AA}^2).$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
N(1)	7808 (4)	2615 (4)	1987 (3)	40 (3)
C(2)	6575 (5)	2324 (5)	1532 (4)	36 (3)
C(3)	7225 (5)	2428 (5)	181 (4)	41 (3)
C(4)	10178 (6)	3094 (5)	-1476 (5)	43 (3)
C(5)	11837 (6)	3524 (7)	-1485 (5)	46 (3)
C(6)	12135 (6)	2883 (5)	-431 (5)	46 (3)
C(7)	10846 (6)	3184 (6)	969 (5)	47 (3)
C(8)	9244 (5)	2885 (5)	919 (4)	41 (3)
C(9)	8915 (6)	2794 (5)	-200 (4)	43 (3)
C(10)	12081 (8)	1315 (7)	-739 (8)	61 (4)
C(11)	13770 (8)	3577 (9)	-431 (8)	73 (5)
C(12)	4855 (6)	2013 (5)	2401 (4)	42 (3)
C(13)	3861 (6)	944 (6)	1987 (5)	50 (3)
C(14)	2240 (6)	670 (7)	2721 (6)	59 (4)
C(15)	1551 (8)	1425 (7)	3878 (6)	66 (5)
C(16)	2528 (8)	2478 (8)	4302 (6)	66 (5)
C(17)	4167 (7)	2791 (6)	3571 (5)	54 (4)
C(18)	7639 (6)	2559 (5)	3331 (4)	41 (3)
C(19)	6992 (6)	1304 (5)	3942 (5)	46 (3)
C(20)	6863 (6)	1241 (6)	5219 (5)	52 (3)
C(21)	7366 (6)	2418 (7)	5891 (5)	57 (4)
C(22)	8025 (6)	3658 (7)	5281 (5)	60 (5)
C(23)	8152 (6)	3751 (5)	3999 (5)	54 (4)
O(1)	9930 (4)	3072 (4)	-2489 (3)	65 (3)

and stereochemical considerations; program *SHELX76* (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and individual isotropic thermal parameters for H atoms gave $R = 0.058$ (unit weights) and $wR = 0.066$ with $w = k\{\sigma(|F_o|)\}^2 + g\{(|F_o|)\}^2\}^{-1}$, $k = 1.8910$ and $g = 0.002780$. Quantity minimized was $\sum w(|F_o| - k|F_c|)^2$. Max. $\Delta/\sigma = 0.293$. Max., min. heights in final difference synthesis 0.22, -0.27 e \AA^{-3} . Atomic scattering factors from *SHELX76*. IBM 370/155 computer.

Discussion. The final atomic coordinates are listed in Table 1 and an *ORTEP* (Johnson, 1965) plot of the molecule is shown in Fig. 1.* The bond lengths and angles involving the non-H atoms are shown in Table 2. Selected torsion angles are given in Table 3. Fig. 2 is a packing diagram of the molecules.

Bond lengths. The bond lengths in the indole fragment of the title compound are in general agreement with corresponding bond lengths in other indoles (Table 4). Intermolecular contacts (Fig. 2) are at van der Waals distances.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, all torsion angles, least-squares planes and intermolecular contact distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43079 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

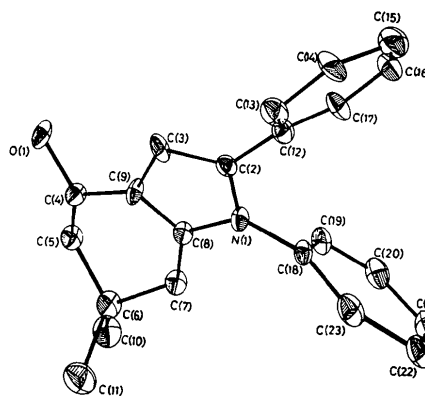


Fig. 1. *ORTEP* plot of the molecule showing the thermal ellipsoids drawn at 50% probability.

Table 2. *Bond lengths* (\AA) and *angles* ($^\circ$) with *e.s.d.'s* in parentheses

C(2)–C(3)	1.372 (6)	C(12)–C(13)	1.399 (8)
C(2)–C(12)	1.483 (7)	C(12)–C(17)	1.374 (6)
C(2)–N(1)	1.397 (7)	C(13)–C(14)	1.378 (7)
C(3)–C(9)	1.431 (7)	C(14)–C(15)	1.353 (8)
C(4)–C(5)	1.511 (8)	C(15)–C(16)	1.386 (10)
C(4)–C(9)	1.440 (6)	C(16)–C(17)	1.393 (8)
C(4)–O(1)	1.230 (7)	C(18)–C(19)	1.385 (7)
C(5)–C(6)	1.543 (10)	C(18)–C(23)	1.388 (7)
C(6)–C(7)	1.551 (6)	C(18)–N(1)	1.437 (6)
C(6)–C(10)	1.510 (8)	C(19)–C(20)	1.377 (8)
C(6)–C(11)	1.532 (9)	C(20)–C(21)	1.377 (8)
C(7)–C(8)	1.482 (8)	C(21)–C(22)	1.373 (9)
C(8)–C(9)	1.379 (8)	C(22)–C(23)	1.386 (8)
C(8)–N(1)	1.378 (4)		
C(2)–C(3)–C(9)	106.4 (3)	C(7)–C(6)–C(11)	108.1 (4)
C(3)–C(2)–C(12)	126.9 (4)	C(10)–C(6)–C(11)	110.2 (5)
C(2)–C(12)–C(13)	119.4 (4)	C(7)–C(8)–C(9)	126.6 (4)
C(2)–C(12)–C(17)	122.6 (4)	C(7)–C(8)–N(1)	125.9 (4)
C(3)–C(2)–N(1)	108.7 (3)	C(9)–C(8)–N(1)	107.5 (3)
C(12)–C(2)–N(1)	124.4 (3)	C(8)–N(1)–C(18)	124.8 (3)
C(2)–N(1)–C(8)	108.9 (3)	C(12)–C(13)–C(14)	121.6 (4)
C(2)–N(1)–C(18)	126.2 (3)	C(13)–C(12)–C(17)	118.0 (4)
C(3)–C(9)–C(4)	130.7 (4)	C(12)–C(17)–C(16)	119.2 (4)
C(3)–C(9)–C(8)	108.6 (3)	C(13)–C(14)–C(15)	121.0 (5)
C(4)–C(5)–C(6)	115.4 (4)	C(14)–C(15)–C(16)	117.9 (5)
C(5)–C(4)–C(9)	115.7 (4)	C(15)–C(16)–C(17)	122.4 (5)
C(4)–C(9)–C(8)	120.7 (4)	C(18)–C(19)–C(20)	119.6 (4)
C(5)–C(4)–O(1)	121.5 (4)	C(19)–C(18)–C(23)	120.2 (4)
C(9)–C(4)–O(1)	122.7 (4)	C(18)–C(23)–C(22)	119.5 (4)
C(5)–C(6)–C(7)	109.7 (4)	C(19)–C(18)–N(1)	119.6 (4)
C(5)–C(6)–C(10)	110.0 (4)	C(23)–C(18)–N(1)	120.2 (4)
C(5)–C(6)–C(11)	109.1 (4)	C(19)–C(20)–C(21)	120.4 (4)
C(6)–C(7)–C(8)	110.0 (4)	C(20)–C(21)–C(22)	120.1 (4)
C(7)–C(6)–C(10)	109.7 (4)	C(21)–C(22)–C(23)	120.2 (4)

Table 3. *Selected torsion angles* ($^\circ$) with *e.s.d.'s* in parentheses

C(4)–C(5)–C(6)–C(7)	-53.6 (6)	N(1)–C(2)–C(12)–C(13)	-138.7 (5)
C(5)–C(6)–C(7)–C(8)	45.5 (6)	N(1)–C(2)–C(12)–C(17)	45.3 (7)
C(6)–C(7)–C(8)–C(9)	-19.5 (7)	C(8)–N(1)–C(18)–C(19)	-115.7 (6)
C(7)–C(8)–C(9)–C(4)	-4.4 (8)	C(8)–N(1)–C(18)–C(23)	62.8 (7)
C(8)–C(9)–C(4)–C(5)	-0.9 (7)	C(8)–C(7)–C(6)–C(11)	164.5 (5)
C(9)–C(4)–C(5)–C(6)	30.6 (7)	C(8)–C(7)–C(6)–C(10)	-75.3 (6)
		C(4)–C(5)–C(6)–C(11)	-171.9 (5)
		C(4)–C(5)–C(6)–C(10)	67.2 (6)

Conjugation. In the chain of bonds linking the pyrrole nitrogen with the O(1) atom, the alternating single and double bonds are respectively shorter and longer than normal, showing that the conjugation between the end atoms of this chain is still present. The lack of activity found by Ramadas, Ramana & Padmanabhan (1978) in other substituted 4-keto tetrahydroindoles may also be attributed to the failure to remove this conjugation.

Conformation. The heterocycle and the substituent phenyl rings are planar. The rotations of the rings [C(12)–C(17) ($\sim 63^\circ$) and C(18)–C(23) ($\sim 45^\circ$)] from the plane of the heterocycle avoid short non-bonded intramolecular contacts C(3)⋯C(13) (3.117 Å), N(1)⋯C(17) (3.141 Å), C(2)⋯C(19) (3.189 Å) and C(8)⋯C(23) (3.160 Å).

While the prevailing conformation of cyclohexene is half chair (Cox, Mkandawire & Mallinson, 1981), the ring C(9)–C(14) assumes a distorted sofa conformation with the asymmetry parameter $\Delta C_s(6) = 8.1(12)^\circ$, as shown by its endocyclic torsion angles (Table 3). Strong *gauche* interactions between the methyl groups at C(6) and the H atoms at C(5) and C(7) are apparently relieved in this conformation. Cyclohexene rings in *cis*-1-acetoxy-3-*tert*-butyl-4-cyanocyclohexene (Viani & Lapasset, 1978) and

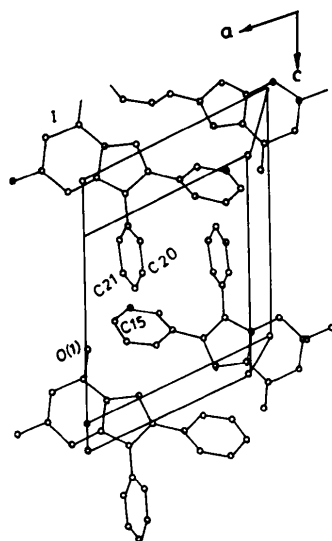


Fig. 2. Packing of molecules in the unit cell. In molecule I the atoms have the coordinates of Table 1. C(20)⋯C(15) = 3.675 and O(1)⋯C(21) = 3.445 Å.

Table 4. Comparison of bond lengths in indoles

Bond type	Nomenclature	Bond distances (Å)			
		A*	B†	C‡	D§
C(sp ²)=C(sp ²)	C(2)–C(3)	1.372 (6)	1.375 (3)	–	1.385 (12)
	C(8)–C(9)	1.379 (8)	1.434 (3)	1.395 (5)	1.383 (13)
C(sp ²)=N	C(2)–N(1)	1.397 (7)	1.392 (2)	1.409 (5)	1.392 (10)
	C(8)–N(1)	1.378 (4)	1.373 (2)	1.351 (5)	1.388 (12)
C(sp ²)–C(sp ²)	C(3)–C(19)	1.431 (7)	1.433 (3)	–	1.442 (11)
	C(4)–C(9)	1.440 (6)	1.401 (3)	–	1.405 (12)

* Title compound.

† 2,3-Diphenylindole (Schmelter, Bradaczek & Luger, 1973).

‡ 1,2,3,9b-Tetrahydro-9bβ-hydroxy-2β-methoxy-1α-phenyl-5H-pyrrolol[1,2-a]isoindol-5-one (Fukuyama, Tanaka & Kakudo, 1980).

§ Quebrachamine (Puglish, Baggio & Baggio, 1976).

trans-3-*tert*-butyl-4-cyano-1-cyclohexenyl acetate (Viani & Lapasset, 1981) and *r*-3-*tert*-butyl-1-4,*c*-5-cyclohexenedicarbonitrile (Cossu, Viani & Lapasset, 1981) assume the sofa conformation.

The authors thank Mr V. Sivanandan, Technical Assistant, and Professor P. T. Manoharan, Head, Regional Sophisticated Instrumentation Centre, IIT Madras, for the use of the X-ray diffractometer.

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