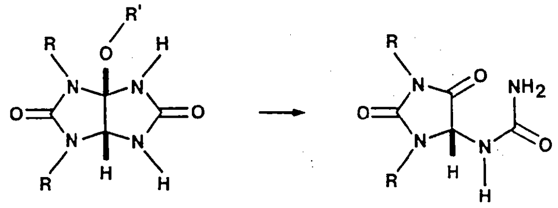
absolute (1*R*,5*R*) configuration of the bicylic ether was assigned with reference to the known configuration of the (−)-menthyl moiety.



(1) (2)

(*a*): R = Me, R' = Menthyl; (*b*): R = R' = H

Related literature. We recently developed a biogenetically modelled route to (−)-(*R*)-1,3-dimethylallantoin (2*a*) *via* regiospecific cleavage of the intermediate (1*a*) bearing the (−)-menthyl ether unit as a chiral auxiliary (Modrić, Drake & Poje, 1989); the key intermediate (1*a*) is the first example of a derivative of the putative bicyclol tautomer (1*b*) of allantoin (2*b*). An X-ray crystal structure determination of (1*a*) was undertaken in order to establish an absolute configurational standard for correlation with (2*a*) and the related naturally occurring homologue (2*b*). The structure of (±)-allantoin, a prototype of (2), has been reported (Mootz, 1965); the tautomerism $(1) \rightleftarrows (2)$ is reminiscent of the formation of cyclol peptides (Cerrini, Fedeli & Mazza, 1971) due to amide–amide interaction.

We thank Professor Giacovazzo for his keen interest and helpful discussions. The financial support of the Research Council of Croatia is also gratefully acknowledged.

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Acta Cryst. (1990). C**46**, 1338–1340

Structure of 4-Piperidone Derivatives. II. 2,6-Bis(*p*-methoxyphenyl)-3,5-dimethyl-4-piperidone

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(*Received* 4 *January* 1990; *accepted* 8 *February* 1990)

Abstract. $C_{21}H_{25}NO_3$, $M_r = 339{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}031\,(2)$, $b = 11{\cdot}675\,(1)$, $c = 22{\cdot}783\,(2)$ Å, $V = 1870{\cdot}19$ Å^3, $Z = 4$, $D_x = 1{\cdot}21$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 6{\cdot}05$ cm^{-1}, $F(000) = 728$, $T = 295$ K, $R = 0{\cdot}046$, $wR = 0{\cdot}050$ for 1817 unique observed reflections $[I > 3\sigma(I)]$. The 4-piperidone ring has a slightly distorted chair conformation, the mean torsion angle being 57·1°; the puckering is enhanced in the area of N(1) and decreased in the area of C(4). The phenyl rings are planar and all molecular dimensions and van der Waals interactions are normal.

Experimental. Crystals were grown from ethanol at room temperature. Data were collected for a

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}(Å^2)
N(1)	0·2200 (3)	0·4841 (1)	0·5973 (1)	0·043 (1)
C(2)	0·1375 (3)	0·4577 (2)	0·5394 (1)	0·046 (1)
C(3)	–0·0707 (4)	0·4151 (2)	0·5492 (1)	0·056 (1)
C(4)	–0·0657 (4)	0·3149 (2)	0·5923 (1)	0·055 (1)
C(5)	0·0450 (3)	0·3325 (2)	0·6482 (1)	0·051 (1)
C(6)	0·2451 (3)	0·3807 (2)	0·6329 (1)	0·045 (1)
C(7)	0·1405 (3)	0·5627 (2)	0·5012 (1)	0·042 (1)
C(8)	0·0771 (3)	0·6684 (2)	0·5214 (1)	0·048 (1)
C(9)	0·0760 (3)	0·7636 (2)	0·4853 (1)	0·049 (1)
C(10)	0·1381 (3)	0·7557 (2)	0·4280 (1)	0·044 (1)
C(11)	0·2037 (3)	0·6516 (2)	0·4067 (1)	0·047 (1)
C(12)	0·2043 (3)	0·5555 (2)	0·4432 (1)	0·047 (1)
O(13)	0·1308 (3)	0·8544 (1)	0·3956 (1)	0·057 (1)
C(14)	0·2098 (4)	0·8527 (2)	0·3383 (1)	0·064 (1)
C(15)	–0·1679 (6)	0·3836 (3)	0·4917 (1)	0·093 (1)
O(16)	–0·1496 (5)	0·2262 (2)	0·5823 (1)	0·090 (1)
C(17)	0·0574 (4)	0·2235 (2)	0·6850 (1)	0·072 (1)
C(18)	0·3589 (3)	0·4073 (2)	0·6874 (1)	0·045 (1)
C(19)	0·5208 (4)	0·3447 (2)	0·7006 (1)	0·051 (1)
C(20)	0·6235 (3)	0·3650 (1)	0·7514 (1)	0·057 (1)
C(21)	0·5683 (3)	0·4503 (2)	0·7899 (1)	0·051 (1)
C(22)	0·4056 (4)	0·5150 (2)	0·7769 (1)	0·054 (1)
C(23)	0·3037 (3)	0·4935 (2)	0·7265 (1)	0·050 (1)
O(24)	0·6601 (3)	0·4779 (2)	0·8402 (1)	0·069 (1)
C(25)	0·8124 (6)	0·4087 (4)	0·8586 (2)	0·101 (1)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

N(1)—C(2)	1·474 (3)	C(12)—C(7)	1·398 (3)
C(2)—C(3)	1·562 (4)	C(5)—C(17)	1·526 (3)
C(3)—C(4)	1·528 (3)	C(4)—O(16)	1·213 (4)
C(4)—C(5)	1·507 (3)	C(6)—N(1)	1·465 (3)
C(5)—C(6)	1·555 (3)	C(6)—C(18)	1·509 (3)
C(2)—C(7)	1·504 (3)	C(18)—C(19)	1·386 (4)
C(7)—C(8)	1·391 (3)	C(19)—C(20)	1·385 (3)
C(8)—C(9)	1·383 (3)	C(20)—C(21)	1·383 (3)
C(9)—C(10)	1·380 (3)	C(21)—C(22)	1·403 (4)
C(10)—C(11)	1·388 (3)	C(22)—C(23)	1·377 (3)
C(11)—C(12)	1·397 (3)	C(23)—C(18)	1·399 (3)
C(10)—O(13)	1·369 (3)	C(21)—O(24)	1·354 (3)
C(3)—C(15)	1·523 (4)	O(24)—C(25)	1·405 (5)
O(13)—C(14)	1·419 (3)		

N(1)—C(2)—C(3)	107·9 (2)	C(12)—C(7)—C(2)	120·2 (2)
C(2)—C(3)—C(4)	108·3 (2)	C(7)—C(2)—C(3)	110·8 (2)
C(3)—C(4)—C(5)	116·8 (2)	C(2)—C(3)—C(15)	112·0 (2)
C(4)—C(5)—C(6)	109·1 (2)	C(15)—C(3)—C(4)	112·2 (2)
C(5)—C(6)—N(1)	108·3 (2)	C(3)—C(4)—O(16)	121·4 (2)
C(6)—N(1)—C(2)	111·8 (2)	O(16)—C(4)—C(5)	121·8 (2)
N(1)—C(2)—C(7)	110·0 (2)	C(5)—C(6)—C(18)	111·7 (2)
C(2)—C(7)—C(8)	121·8 (2)	C(6)—C(18)—C(19)	120·4 (2)
C(7)—C(8)—C(9)	121·2 (2)	C(18)—C(19)—C(20)	121·3 (2)
C(8)—C(9)—C(10)	120·5 (2)	C(19)—C(20)—C(21)	120·5 (2)
C(9)—C(10)—C(11)	119·7 (2)	C(20)—C(21)—C(22)	118·9 (2)
C(10)—C(11)—C(12)	119·8 (2)	C(21)—C(22)—C(23)	120·2 (2)
C(11)—C(12)—C(7)	120·9 (2)	C(23)—C(18)—C(6)	121·7 (2)
N(1)—C(6)—C(18)	110·5 (2)	C(10)—O(13)—C(14)	118·0 (2)
C(11)—C(10)—O(13)	124·1 (2)	C(21)—O(24)—C(25)	118·6 (3)
C(9)—C(10)—O(13)	116·2 (2)	C(22)—C(21)—O(24)	116·1 (2)
C(22)—C(23)—C(18)	121·2 (2)	C(20)—C(21)—O(24)	125·1 (2)
C(19)—C(18)—C(23)	118·0 (2)	C(4)—C(5)—C(17)	112·3 (2)
C(6)—C(5)—C(17)	111·9 (2)		

N(1)—C(2)—C(3)—C(4)	− 54·8 (2)	C(4)—C(5)—C(6)—N(1)	54·6 (2)
C(2)—C(3)—C(4)—C(5)	49·7 (3)	C(5)—C(6)—N(1)—C(2)	− 66·8 (2)
C(3)—C(4)—C(5)—C(6)	− 49·5 (3)	C(6)—N(1)—C(2)—C(3)	67·2 (2)

colourless transparent crystal (0·20 × 0·20 × 0·25 mm) with an Enraf–Nonius CAD-4 diffractometer using Ni-filtered Cu $K\alpha$ radiation. Unit-cell parameters were derived from a least-squares analysis of 25 reflections with $25 \leq 2\theta \leq 35°$. Intensity data were collected with the ω–2θ scan technique, 1986 reflections (h, $0\rightarrow 8$; k, $0\rightarrow 13$; l, $0\rightarrow 25$) up to $\theta = 65°$ were measured, of which 1817 had intensities greater than $3\sigma(I)$. During data collection three standard reflections, monitored after every 2 h of X-ray exposure, indicated no decay over the full 26 h period. The intensity data were corrected for Lp and for absorption ($T_{min} = 0{\cdot}9512$ and $T_{max} = 0{\cdot}9963$). From the observed systematic absences the space group is found to be $P2_12_12_1$. The structure was solved by direct methods using *SHELX*86 (Sheldrick, 1986) and refined on F by weighted full-matrix least

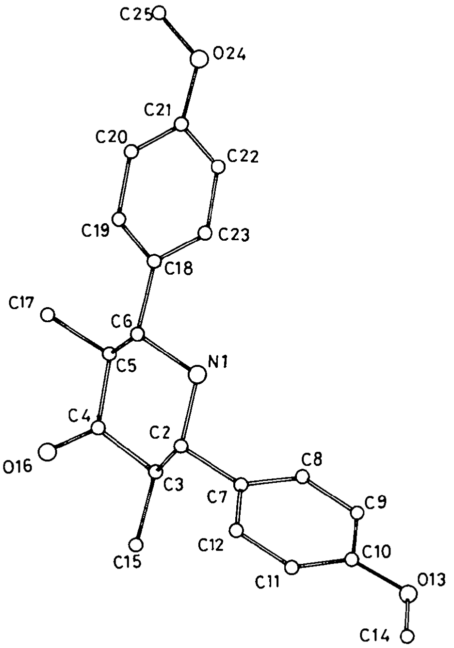

Fig. 1. A view of the molecule with the atomic numbering scheme.

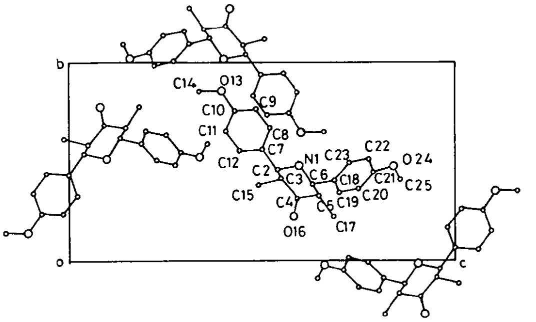

Fig. 2. Packing diagram viewed down the a axis.

squares on a MicroVAX II computer with *SHELX76* (Sheldrick, 1976). H atoms were located from a difference Fourier synthesis. All the H atoms were allowed to refine isotropically in final cycles. Final maximum $\Delta/\sigma = 0{\cdot}05$. Maximum and minimum heights in final difference Fourier synthesis 0·15 and $-0{\cdot}22\ \text{e Å}^{-3}$ respectively. Refinement with weights given by $w = 1{\cdot}0000/[\sigma^2(F) + 0{\cdot}010894(F_o)^2]$ converged at $R = 0{\cdot}046$, $wR = 0{\cdot}050$. Atomic scattering factors were those of *SHELX*. Final positional and thermal parameters are listed in Table 1 and bond lengths and angles in Table 2.* A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule showing the molecular geometry is presented in Fig. 1 and the molecular packing in the unit cell in Fig. 2.

Related literature. The 4-piperidone ring in this structure has a slightly distorted chair conformation; puckering is enhanced in the area of N(1) and decreased in the area of C(4). A similar conformational feature is also observed in the 4-piperidone rings of 3-methyl-2,6-diphenyl-4-piperidone (Sekar, Parthasarathy & Rajalingam, 1990) and 1,1′-di-(4-pyridyl)-2,2′,6,6′-bi(4-piperidone) dihydrochloride dihydrate (Cheer, Cosgrove & Vittimberga, 1984).

Thanks are due to the University Grants Commission, India, for the award of JRF under Special Assistance Programme to KS.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52698 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). C**46**, 1340–1342

Structure of an Oxazoylphenyloxazole

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(*Received* 31 *October* 1989; *accepted* 31 *January* 1990)

Abstract. 4-(3-Methylphenyl)-5-phenyl-2-[4-(5-phenyl-2-oxazolyl)phenyl]oxazole, $C_{31}H_{22}N_2O_2$, $M_r = 454{\cdot}53$, triclinic, $P\bar{1}$, $a = 7{\cdot}649\ (1)$, $b = 9{\cdot}824\ (1)$, $c = 15{\cdot}928\ (1)$ Å, $\alpha = 95{\cdot}36\ (1)$, $\beta = 100{\cdot}43\ (1)$, $\gamma = 94{\cdot}13\ (1)°$, $V = 1167{\cdot}12$ Å^3, $Z = 2$, $D_x = 1{\cdot}293$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 476$, $T = 298$ K, $R = 0{\cdot}043$ for 3498 observed reflections. The structure was determined to investigate the planarity of the ring system. The rings were found to be coplanar (torsional angles between rings 5° or less), except for the rings substituted in positions C(4) and C(5) where dihedral angles of 22·4 (2) and 34·1 (2)° were found.

Experimental. The synthetic compound was crystallized by liquid–vapor diffusion from a solution in a mixture of $CHCl_3$ and toluene with *n*-hexane. The crystal dimensions were 0·3 × 0·7 × 0·75 mm. Data were collected on a Stoe–Siemens four-circle diffractometer with monochromated Mo $K\alpha$ radiation using the profile-fitting mode involving variable scan

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