Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

N(1)—N(2)	1·409 (8)	N(1)—C(12)	1·328 (9)
N(2)—C(3)	1·380 (9)	N(2)—C(15)	1·411 (9)
C(3)—C(13)	1·417 (10)	C(3)—O(14)	1·265 (9)
C(4)—N(5)	1·336 (9)	C(4)—C(13)	1·363 (10)
N(5) C(11)	1·399 (9)	C(6) C(7)	1·385 (10)
C(6)—C(11)	1·388 (10)	C(7)—C(8)	1·375 (11)
C(8)—C(9)	1·376 (11)	C(9)—C(10)	1·409 (10)
C(10)—C(11)	1·409 (10)	C(10)—C(12)	1·437 (10)
C(12)—C(13)	1·441 (10)	C(15)—S(16)	1·705 (7)
C(15)—C(19)	1·386 (10)	S(16)—C(17)	1·724 (7)
C(17)—C(18)	1·336 (11)	C(17)—C(20)	1·506 (11)
C(18)—C(19)	1·411 (11)		
N(2)—N(1)—C(12)	103·2 (5)	N(1)—N(2)—C(3)	114·5 (5)
N(1)—N(2)—C(15)	119·0 (5)	C(3)—N(2)—C(15)	126·4 (6)
N(2)—C(3)—C(13)	103·6 (6)	N(2)—C(3)—O(14)	124·5 (6)
C(13)—C(3)—O(14)	131·9 (7)	N(5)—C(4)—C(13)	118·8 (6)
C(4)—N(5)—C(11)	123·5 (6)	C(7)—C(6)—C(11)	118·9 (6)
C(6)—C(7)—C(8)	120·9 (7)	C(7)—C(8)—C(9)	121·0 (7)
C(8)—C(9)—C(10)	119·8 (7)	C(9)—C(10)—C(11)	118·3 (6)
C(9)—C(10)—C(12)	124·9 (6)	C(11)—C(10)—C(12)	116·8 (6)
N(5)—C(11)—C(6)	118·5 (6)	N(5)—C(11)—C(10)	120·4 (6)
C(6)—C(11)—C(10)	121·1 (6)	N(1)—C(12)—C(10)	129·0 (6)
N(1)—C(12)—C(13)	112·3 (6)	C(10)—C(12)—C(13)	118·7 (6)
C(3)—C(13)—C(4)	131·8 (7)	C(3)—C(13)—C(12)	106·3 (6)
C(4)—C(13)—C(12)	121·8 (6)	N(2)—C(15)—S(16)	120·5 (5)
N(2)—C(15)—C(19)	127·8 (6)	S(16)—C(15)—C(19)	111·7 (5)
C(15)—S(16)—C(17)	91·3 (3)	S(16)—C(17)—C(18)	112·0 (6)
S(16)—C(17)—C(20)	121·5 (5)	C(18)—C(17)—C(20)	126·5 (7)
C(17)—C(18)—C(19)	113·5 (7)	C(15)—C(19)—C(18)	111·4 (7)

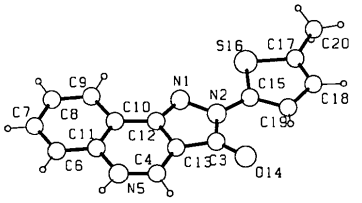

Fig. 1. Perspective view of the title compound, with the atom-numbering system.

factors were calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1, 2, 3, 4$) (*International Tables for X-ray Crystallography*, 1974). Calculations were performed on a FACOM M340R computer at Shionogi Research Laboratories. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond distances and angles are listed in Table 2.* A perspective view of the molecule with the atom-numbering system, drawn using the program *PLUTO* (Motherwell & Clegg, 1978), is presented in Fig. 1.

Related literature. The structure of the title compound has been discussed by Shindo, Takada, Murata, Eigyo & Matsushita (1989).

* Lists of H-atom coordinates, anisotropic temperature factors of the non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52627 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4-Piperidone Derivatives. I. 3-Methyl-2,6-diphenyl-4-piperidone

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Abstract. $C_{18}H_{19}NO$, $M_r = 265{\cdot}3$, monoclinic, $C2/c$, $a = 19{\cdot}266\ (2)$, $b = 6{\cdot}999\ (2)$, $c = 21{\cdot}653\ (1)$ Å, $\beta = 94{\cdot}42\ (1)°$, $V = 2911{\cdot}07$ Å^3, $Z = 8$, $D_x = 1{\cdot}21$ g cm^{-3}, λ(Cu $K\alpha$) $= 1{\cdot}5418$ Å, $\mu = 5{\cdot}05$ cm^{-1}, $F(000) = 1136$, $T = 295$ K, $R = 0{\cdot}047$ for 1655 unique observed reflections [$I > 3\sigma(I)$]. The 4-piperidone has

* DCB contribution No. 748.

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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)	0·6262 (1)	0·1452 (2)	0·4698 (1)	0·047 (1)
C(2)	0·6700 (1)	0·0457 (3)	0·4270 (1)	0·049 (1)
C(3)	0·6439 (1)	−0·1621 (3)	0·4180 (1)	0·060 (1)
C(4)	0·6434 (1)	−0·2546 (3)	0·4805 (1)	0·060 (1)
C(5)	0·6073 (1)	−0·1444 (3)	0·5280 (1)	0·061 (1)
C(6)	0·6331 (1)	0·0634 (3)	0·5323 (1)	0·049 (1)
C(7)	0·6678 (1)	0·1474 (3)	0·3652 (1)	0·047 (1)
C(8)	0·6058 (1)	0·1776 (4)	0·3301 (1)	0·061 (1)
C(9)	0·6053 (1)	0·2647 (4)	0·2718 (1)	0·069 (1)
C(10)	0·6665 (2)	0·3209 (4)	0·2493 (1)	0·069 (1)
C(11)	0·7279 (1)	0·2930 (4)	0·2842 (1)	0·070 (1)
C(12)	0·7286 (1)	0·2052 (4)	0·3418 (1)	0·056 (1)
C(13)	0·6852 (1)	−0·2755 (4)	0·3737 (1)	0·088 (1)
O(14)	0·6701 (1)	−0·4101 (2)	0·4923 (1)	0·074 (1)
C(15)	0·5927 (1)	0·1739 (3)	0·5774 (1)	0·047 (1)
C(16)	0·5959 (1)	0·1174 (4)	0·6389 (1)	0·063 (1)
C(17)	0·5590 (1)	0·2125 (4)	0·6817 (1)	0·068 (1)
C(18)	0·5189 (1)	0·3678 (3)	0·6642 (1)	0·063 (1)
C(19)	0·5161 (1)	0·4284 (4)	0·6040 (1)	0·061 (1)
C(20)	0·5527 (1)	0·3330 (3)	0·5608 (1)	0·050 (1)

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

N(1)—C(2)	1·475 (3)	C(12)—C(7)	1·373 (3)
C(2)—C(3)	1·546 (3)	C(3)—C(13)	1·517 (4)
C(3)—C(4)	1·501 (3)	C(4)—O(14)	1·222 (3)
C(4)—C(5)	1·499 (3)	C(6)—N(1)	1·466 (3)
C(5)—C(6)	1·538 (3)	C(6)—C(15)	1·508 (3)
C(2)—C(7)	1·513 (3)	C(15)—C(16)	1·386 (3)
C(7)—C(8)	1·382 (3)	C(16)—C(17)	1·382 (3)
C(8)—C(9)	1·401 (3)	C(17)—C(18)	1·370 (3)
C(9)—C(10)	1·368 (4)	C(18)—C(19)	1·368 (3)
C(10)—C(11)	1·368 (4)	C(19)—C(20)	1·386 (3)
C(11)—C(12)	1·390 (3)	C(20)—C(15)	1·386 (3)
N(1)—C(2)—C(3)	109·1 (2)	C(12)—C(7)—C(2)	119·9 (2)
C(2)—C(3)—C(4)	108·5 (2)	C(7)—C(2)—C(3)	110·1 (2)
C(3)—C(4)—C(5)	115·6 (2)	C(2)—C(3)—C(13)	113·0 (2)
C(4)—C(5)—C(6)	111·3 (2)	C(13)—C(3)—C(4)	112·6 (2)
C(5)—C(6)—N(1)	107·9 (2)	C(3)—C(4)—O(14)	122·7 (2)
C(6)—N(1)—C(2)	112·3 (2)	O(14)—C(4)—C(5)	121·7 (2)
N(1)—C(2)—C(7)	110·8 (2)	C(5)—C(6)—C(15)	110·1 (2)
C(2)—C(7)—C(8)	121·5 (2)	C(6)—C(15)—C(16)	119·3 (2)
C(7)—C(8)—C(9)	120·5 (2)	C(15)—C(16)—C(17)	121·3 (2)
C(8)—C(9)—C(10)	120·0 (2)	C(16)—C(17)—C(18)	120·3 (2)
C(9)—C(10)—C(11)	119·7 (2)	C(17)—C(18)—C(19)	119·3 (2)
C(10)—C(11)—C(12)	120·5 (2)	C(18)—C(19)—C(20)	120·6 (2)
C(11)—C(12)—C(7)	120·8 (2)	C(20)—C(15)—C(6)	123·3 (2)
N(1)—C(6)—C(15)	112·5 (2)		
N(1)—C(2)—C(3)—C(4)	−55·3 (2)	C(4)—C(5)—C(6)—N(1)	53·3 (2)
C(2)—C(3)—C(4)—C(5)	50·5 (2)	C(5)—C(6)—N(1)—C(2)	−62·7 (2)
C(3)—C(4)—C(5)—C(6)	−50·4 (3)	C(6)—N(1)—C(2)—C(3)	65·0 (2)

a slightly distorted chair conformation, the mean torsion angle being 56·2°; 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 colourless transparent crystal (0·20 × 0·20 × 0·30 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 $25 \le 2\theta \le 35°$. Intensity data were collected with the ω–2θ scan technique, 2003 reflections ($h -21 \rightarrow 21$, $k 0 \rightarrow 7$, $l 0 \rightarrow 24$) up to $\theta = 60°$ were measured, of which 1655 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 25 h period. The intensity data were corrected for Lp and for absorption ($T_{min} = 0·9570$ and $T_{max} = 0·9984$). From the observed systematic absences and the statistical test for a centre of symmetry the space group is $C2/c$. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986) and refined on F by weighted full-matrix least squares on a MicroVAX II computer with *SHELX*76 (Sheldrick, 1976). The hydrogen atoms were located from a difference Fourier map except C2H, C5H and C8H which were fixed geometrically. All hydrogen atoms were allowed to refine isotropically in final cycles. Final maximum $\Delta/\sigma = 0·06$. Maximum and minimum heights in final difference Fourier synthesis = 0·16 and $-0·18$ e Å^{-3} respectively. Refinement with weights given by $w = 1·0000/[\sigma^2(F) + 0·000134(F_o)^2]$ converged at $R = 4·7\%$ ($wR = 5·0\%$). Atomic scattering factors were those of *SHELX*. Final atomic parameters are listed in Table 1 and bond lengths

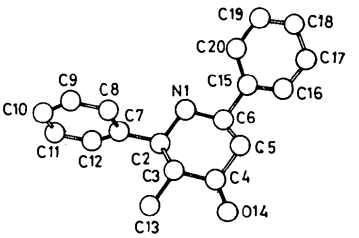

Fig. 1. A view of the molecule with atom numbering.

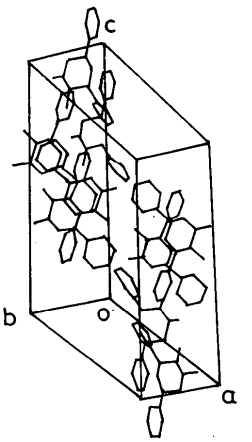

Fig. 2. Packing diagram.

and angles in Table 2.* A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule showing the molecular geometry is presented in Fig. 1, molecular packing in the unit cell in Fig. 2.

Related literature. The 4-piperidone 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 3,5-dimethyl-2,6-di(*p*-methoxyphenyl)-4-piperidone (Sekar, Parthasarathy & Radhakrishnan, 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.

* 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 52599 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dendrobine Support Studies. The Structure of a Novel 3-Azatricyclo[6.2.1.$0^{4,11}$]-undecane Derivative

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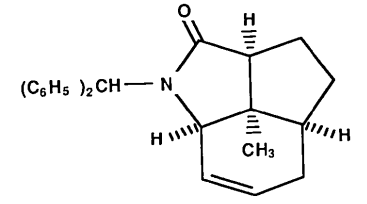
Abstract. (1*S**,4*R**,8*S**,11*R**)-3-Diphenylmethyl-11-methyl-3-azatricyclo[6.2.1.$0^{4,11}$]undec-5-en-2-one, $C_{24}H_{25}NO$, $M_r = 343{\cdot}47$, monoclinic, $P2_1$, $a = 10{\cdot}248\ (3)$, $b = 8{\cdot}859\ (2)$, $c = 10{\cdot}344\ (2)$ Å, $\beta = 99{\cdot}816\ (14)°$, $V = 925{\cdot}4\ (3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}23$ g cm^{-1} (163 K), λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}6927$ cm^{-1}, $F(000) = 368$, $T = 163$ K, $R = 0{\cdot}0423$ for 2569 reflections. The compound is spontaneously resolved upon crystallization. The N atom appears to be sp^2 hybridized [N is 0·0901 (13) Å from plane through three ligand atoms] and in conjugation with the carbonyl group [short N—C bond 1·351 (2) Å]. The tricyclic ring system is concave. Ring strain appears to affect C—C bond lengths of the central atom of the 3-ring system. The average C—C bond length for this atom to other ring atoms is 1·551 (2) while the average for all other sp^3 C—sp^3 C bonds is 1·530 (2) Å.

Experimental. (1) was synthesized by an intramolecular Diels–Alder reaction of the corresponding trienamide. Details of the synthetic procedure have been published elsewhere (Martin & Li, 1989). Colorless crystals of (1) (m.p. 423–424 K) were



(1)

obtained by slow evaporation from ethyl acetate–hexanes (1:19) from which (1) resolves spontaneously. The data crystal was cut from a large block and had approximate dimensions 0·35 × 0·37 × 0·45 mm. The data were collected on a Syntex $P2_1$ diffractometer, with a graphite monochromator, and equipped with a Syntex LT-1 low temperature delivery system (163 K). Lattice parameters were obtained from the least-squares refinement of 45 reflections with $24{\cdot}2 < 2\theta < 31{\cdot}2°$. Data were collected using the ω-scan

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