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).

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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 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

By V. M. Lynch, W. Li,† S. F. Martin and B. E. Davis

Department of Chemistry, University of Texas at Austin, Austin, Texas 78712, *USA*

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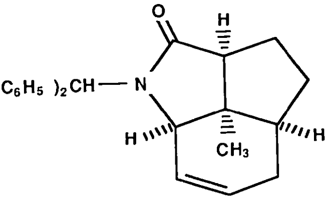
Abstract. (1S*,4R*,8S*,11R*)-3-Diphenylmethyl-11-methyl-3-azatricyclo[6.2.1.$0^{4,11}$]undec-5-en-2-one, $C_{24}H_{25}NO$, M_r = 343·47, monoclinic, $P2_1$, a = 10·248 (3), b = 8·859 (2), c = 10·344 (2) Å, β = 99·816 (14)°, V = 925·4 (3) Å^3, Z = 2, D_x = 1·23 g cm^{-1} (163 K), λ(Mo $K\alpha$) = 0·7107 Å, μ = 0·6927 cm^{-1}, F(000) = 368, T = 163 K, R = 0·0423 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 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 24·2 < 2θ < 31·2°. Data were collected using the ω-scan



(1)

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† Permanent address: Department of Chemistry, Nankai University, Tianjin, People's Republic of China.

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

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

	x	y	z	U_{eq}(Å^2)
C1	0·0582 (2)	0·6110 (3)	0·7866 (2)	0·0290 (6)
C2	0·1699 (2)	0·6701 (3)	0·7220 (2)	0·0255 (6)
N3	0·25982 (14)	0·5586	0·71926 (14)	0·0204 (5)
C4	0·2304 (2)	0·4173 (3)	0·7830 (2)	0·0205 (5)
C5	0·2336 (2)	0·2805 (3)	0·6980 (2)	0·0237 (6)
C6	0·1417 (2)	0·1746 (3)	0·6822 (2)	0·0299 (6)
C7	0·0187 (2)	0·1873 (4)	0·7402 (3)	0·0434 (8)
C8	−0·0183 (2)	0·3500 (3)	0·7623 (2)	0·0346 (7)
C9	−0·0750 (2)	0·4395 (4)	0·6394 (2)	0·0398 (8)
C10	−0·0736 (2)	0·6007 (4)	0·6896 (3)	0·0396 (8)
C11	0·0969 (2)	0·4480 (3)	0·8320 (2)	0·0259 (6)
C12	0·1175 (3)	0·4280 (4)	0·9810 (2)	0·0398 (8)
O13	0·17868 (15)	0·7973 (2)	0·6797 (2)	0·0364 (5)
C14	0·3892 (2)	0·5890 (3)	0·6802 (2)	0·0210 (5)
C15	0·3969 (2)	0·5377 (3)	0·5411 (2)	0·0221 (5)
C16	0·2857 (2)	0·4910 (3)	0·4548 (2)	0·0261 (6)
C17	0·2940 (2)	0·4502 (3)	0·3265 (2)	0·0312 (6)
C18	0·4149 (2)	0·4560 (3)	0·2838 (2)	0·0315 (7)
C19	0·5260 (2)	0·5028 (3)	0·3684 (2)	0·0323 (7)
C20	0·5178 (2)	0·5455 (3)	0·4956 (2)	0·0278 (6)
C21	0·4995 (2)	0·5318 (3)	0·7872 (2)	0·0229 (5)
C22	0·5665 (2)	0·3972 (3)	0·7773 (2)	0·0270 (6)
C23	0·6603 (2)	0·3463 (3)	0·8826 (2)	0·0340 (7)
C24	0·6868 (2)	0·4300 (3)	0·9968 (2)	0·0387 (7)
C25	0·6198 (2)	0·5634 (3)	1·0071 (2)	0·0377 (7)
C26	0·5266 (2)	0·6140 (3)	0·9035 (2)	0·0300 (6)

technique (5718 reflections, 2859 unique, R_{int} = 0·0264 from averaging symmetry equivalent reflections), with a 2θ range from 4·0–60°, using a 1° ω scan at 6–12° min^{-1} ($h = -14\rightarrow17$, $k = 0\rightarrow6$, $l = -14\rightarrow14$). Four reflections ($\bar{3}\bar{1}2$; $00\bar{2}$; 200; 122) were remeasured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was 1·3%, Henslee & Davis, 1975). The data were also corrected for Lp effects and absorption (based on crystal shape; transmission factor range 0·9757–0·9805). The data reduction routine is described in Riley & Davis (1976). Reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (290 reflections). The structure was solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by full-matrix least-squares procedures (Sheldrick, 1976) with anisotropic thermal parameters for the non-H atoms. H atoms were obtained from a ΔF map and refined with isotropic thermal parameters. A total of 334 parameters were refined. The y coordinate of N3 was fixed to define the origin. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{peak} - I_{background}) \times$ (scan rate); 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects, absorption and decay. $\sigma(I)$ was estimated from couting statistics; $\sigma(I) = [(I_{peak} + I_{background})^{1/2} \times$ (scan rate)]. The final R =

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

1	2	3	1—2	1—2—3
C2	C1	C10	1·514 (3)	112·2 (2)
C10	C1	C11	1·542 (3)	107·0 (2)
C11	C1	C2	1·550 (4)	106·4 (2)
N3	C2	O13	1·355 (2)	125·1 (2)
N3	C2	C1		109·0 (2)
O13	C2	C1	1·217 (3)	125·9 (2)
C4	N3	C14	1·470 (2)	122·76 (13)
C4	N3	C2		114·8 (2)
C14	N3	C2	1·475 (2)	121·26 (14)
C5	C4	C11	1·501 (3)	116·0 (2)
C5	C4	N3		113·4 (2)
C11	C4	N3	1·561 (3)	104·8 (2)
C6	C5	C4	1·320 (3)	124·0 (2)
C7	C6	C5	1·490 (4)	122·1 (2)
C8	C7	C6	1·517 (4)	112·5 (2)
C9	C8	C11	1·527 (4)	104·5 (2)
C9	C8	C7		115·9 (2)
C11	C8	C7	1·541 (3)	114·5 (2)
C10	C9	C8	1·519 (5)	102·9 (2)
C1	C10	C9		103·6 (2)
C12	C11	C1	1·530 (3)	113·4 (2)
C12	C11	C4		109·2 (2)
C12	C11	C8		111·7 (2)
C1	C11	C4		104·9 (2)
C1	C11	C8		104·3 (2)
C4	C11	C8		113·2 (2)
C15	C14	C21	1·524 (3)	115·8 (2)
C15	C14	N3		113·45 (15)
C21	C14	N3	1·528 (3)	109·2 (2)
C16	C15	C20	1·385 (3)	118·4 (2)
C16	C15	C14		121·9 (2)
C20	C15	C14	1·400 (3)	119·6 (2)
C17	C16	C15	1·392 (3)	121·0 (2)
C18	C17	C16	1·385 (3)	119·8 (2)
C19	C18	C17	1·377 (3)	119·8 (2)
C20	C19	C18	1·386 (3)	120·5 (2)
C15	C20	C19		120·4 (2)
C22	C21	C26	1·388 (3)	119·0 (2)
C22	C21	C14		123·0 (2)
C26	C21	C14	1·393 (3)	117·8 (2)
C23	C22	C21	1·399 (3)	120·1 (2)
C24	C23	C22	1·382 (4)	120·1 (2)
C25	C24	C23	1·380 (4)	119·8 (2)
C26	C25	C24	1·383 (3)	120·3 (2)
C21	C26	C25		120·6 (2)

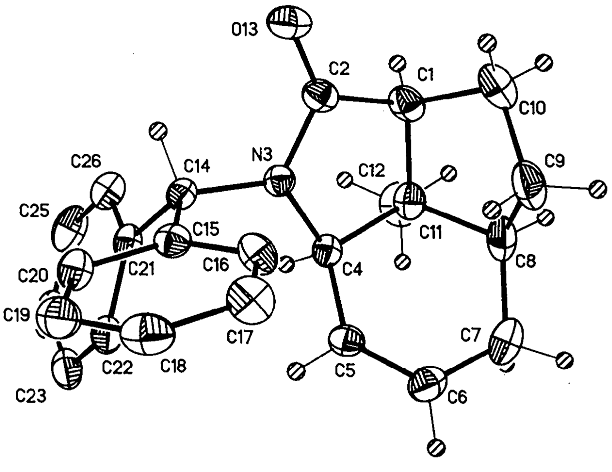

Fig. 1. View of (1) showing the atomic labelling scheme. Thermal ellipsoids are scaled to the 50% probability level. Phenyl ring H atoms are omitted for clarity. H atoms are scaled to an arbitrary size.

0·0423 for 2569 reflections, $wR = 0·0351$ ($R_{\rm all} = 0·0492$, $wR_{\rm all} = 0·0357$) and a goodness of fit = 1·892. The maximum $|\Delta/\sigma| < 0·1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were −0·20 and 0·30 e Å^{-3}, respectively. Differentiation between enantiomorphs was not possible on the basis of the X-ray diffraction results (wR for enantiomorph was 0·0351). The scattering factors for the non-H atoms were obtained from Cromer & Mann (1968), with anomalous-dispersion corrections from the work of Cromer & Liberman (1970), while scattering factors for the H atoms were taken from Stewart, Davidson & Simpson (1965). The linear absorption coefficient was calculated using values in *International Tables for X-ray Crystallography* (1974). Atomic positional and thermal parameters for the non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2.* The atomic labelling scheme is shown in Fig. 1. Fig. 1 was generated using the Nicolet XRD *SHELXTL-PLUS* software package (Sheldrick, 1987). The least-squares-planes program was supplied by Cordes (1983); other computer programs from reference 11 of Gadol & Davis (1982).

Related literature. The crystal structure of the α-phenylethyl analog of (1) has been previously reported and references cited therein (Lynch, Li & Martin, 1988).

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* Tables of anisotropic thermal parameters, positional and thermal parameters for the H atoms, bond distances and angles involving the H atoms, torsion angles, least-squares planes, structure-factor amplitudes and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52597 (28 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 1,2-Bis(2-methyl-4-quinazolinyl)ethylene

By Wolfgang Hiller and Mehmet Akkurt*

Institut für Anorganische Chemie der Universität Tübingen, Auf der Morgenstelle 18, *D*-7400 *Tübingen,*
Federal Republic of Germany

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Abstract. $C_{20}H_{16}N_4$, $M_r = 312·38$, monoclinic, $P2_1/n$, $a = 9·654\,(1)$, $b = 7·544\,(1)$, $c = 11·018\,(1)$ Å, $\beta = 99·84\,(1)°$, $V = 790·6$ Å^3, $Z = 2$, $D_x = 1·312$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·54184$ Å, $\mu = 5·964$ cm^{-1}, $F(000) = 328$, $T = 293$ K. The final R value converged to 0·049 for 907 significant [$I > 3\sigma(I)$] reflections. In the asymmetric unit is a half molecule completed by an inversion center in the ethylene bond. The resulting planar molecule is the *trans* isomer.

Experimental. The product was obtained by oxidation of 2,4-dimethylquinazoline with SeO_2 (Kepez, 1989). To clarify which of the methyl groups is oxidated we decided to perform a structure determination. A yellow single crystal of approximate dimensions 0·40 × 0·15 × 0·25 mm was mounted on a glass fiber. The systematic absences indicated space

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* Permanent address: Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey.

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