(Tables 1 and 2).* Fig. 2 shows a view of the molecular packing of the bases; the ribose and acetyl atoms are omitted for clarity.

**Related literature.** The conformational parameters used follow the guidelines of the IUPAC–IUB Joint Commission on Biochemical Nomenclature (1983). The related compound 2′,3′,5′-tri-*O*-acetylguanosine has been studied recently (Wilson, Low & Tollin, 1985; Low, Tollin, Wilson & Scrimgeour, 1986). Conformational parameters for nucleosides are discussed by Saenger (1984).

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* Lists of structure factors,, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44344 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Structure of the *ABC* Ring Synthon of Dendrobine

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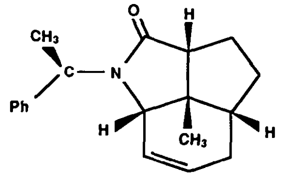
**Abstract.** (1*R*,4*S*,8*S*,11*R*)-11-Methyl-3-[(*R*)-α-phenylethyl]-3-azatricyclo[6.2.1.$0^{4,11}$]undec-5-en-2-one, $C_{19}H_{23}NO$, $M_r = 281{\cdot}40$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}534\,(2)$, $b = 11{\cdot}055\,(4)$, $c = 14{\cdot}372\,(4)$ Å, $V = 1514{\cdot}8\,(8)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}23$ (163 K), $D_m = 1{\cdot}15$ g cm$^{-3}$ (295 K), $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}702$ cm$^{-1}$, $F(000) = 608$, $T = 163$ K, $R = 0{\cdot}0387$ for 2310 reflections. The absolute configuration is assigned on the basis of internal comparison with the α-methylbenzylamine moiety. The tricyclic ring system is concave. The N appears to be $sp^2$ hybridized [N is 0·0661 (1) Å from the plane through C atoms bound to it] and conjugated through the carbonyl group {short C—N bond [1·351 (2) Å]}. Ring strain appears to cause a slight increase in the C—C bond lengths of the center atom of the tricyclic ring system. The average bond length from this atom to the three other ring C atoms is 1·557 (1) Å while the average bond length for all other $sp^3$-C to $sp^3$-C bonds is 1·529 (1) Å.

**Experimental.** The title compound (1) was synthesized by an intramolecular Diels–Alder reaction of the corresponding trienamide which was heated in a sealed tube in xylene. Details of the experimental procedure will be reported elsewhere (Martin & Li, 1987).



(1)

(*R*)-(+)-α-Methylbenzylamine, upon which the assignment of absolute configuration was made, was purchased from the Aldrich Chemical Company. Colorless block, cut from a larger crystal, 0·49 × 0·61 × 0·87 mm, from 1:3 ethyl acetate–hexane. Crystal density by flotation method in aqueous $ZnCl_2$. Syntex $P2_1$ diffractometer, graphite monochromator with Syntex LT-1 low-temperature delivery system (163 K). Lattice parameters from least-squares refinement of 30 reflections with $25{\cdot}5 < 2\theta < 31{\cdot}5°$. ω-scan technique (5018 reflections, 2516 unique, $R_{\text{int}} = 0{\cdot}0294$ from merging $hkl$ and $\bar{h}\bar{k}l$ reflections; $h\ -13\rightarrow15$;

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$k$ −15→15; $l$ 0→20), $2\theta$ range 4·0–60·0°, 1·0° $\omega$ scan at 6–12° min$^{-1}$. Four reflections ($\bar{1}\bar{2}\bar{3}$, 002, 040, 200) were remeasured every 96 reflections to monitor instrument and crystal stability. Analysis of these data indicated that no corrections for crystal decay or instrument instability were necessary (Henslee & Davis, 1975). Data were corrected for Lp effects and absorption (based on crystal shape; transmission-factor range 0·946–0·973). Data reduction described in Riley & Davis (1976). Reflections having $F_o < 4\sigma(F_o)$ considered unobserved (206 reflections). Structure solved by direct methods (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. 14 of the H-atom positions from a $\Delta F$ map, the remaining nine were calculated in ideal positions and all were refined with isotropic thermal parameters. During the latter stages of refinement the positions of 16 H atoms (H3, H5, H6, H8*A*, H8*B*, H9, H10*A*, H10*B*, H11*A*, H11*B*, H14, H17, H18, H19, H20 and H21) deviated significantly from expected geometry. These atoms were fixed in ideal positions during the final refinement cycles. 234 parameters refined. $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0{\cdot}5kI^{-1/2} \times \{[\sigma(I)]^2 + (0{\cdot}04I)^2\}^{1/2}$. Intensity $I$ is given by $(I_{\rm peak} - I_{\rm background}) \times$ (scan rate), 0·04 is a factor to downweight intense reflections and to account for instrument instability and $k$ is the correction due to Lp effects and absorption. $\sigma(I)$ estimated from counting statistics; $\sigma(I) = (I_{\rm peak} + I_{\rm background})^{1/2} \times$ (scan rate). Final $R$ = 0·0387 for 2310 reflections, $wR = 0{\cdot}0427$ ($R_{\rm all}$ = 0·0435, $wR_{\rm all} = 0{\cdot}0431$) and a goodness of fit, $S$ = 1·821. Maximum $|\Delta/\sigma| = 0{\cdot}14$ in the final refinement cycle and the minimum and maximum peaks in the final $\Delta F$ map were −0·13 and 0·20 e Å$^{-3}$, respectively. The absolute configuration of (1) was assigned by internal comparison with that at C14 of the α-methylbenzylamine moiety which was determined by Bush, Dullforce & Sim (1969). The enantiomorph refined to $wR = 0{\cdot}0427$ and, therefore, discrimination between enantiomers could not be made on the basis of X-ray diffraction results. Scattering factors for the non-H atoms from Cromer & Mann (1968), with anomalous-dispersion corrections from Cromer & Liberman (1970), while scattering factors for the H atoms were from Stewart, Davidson & Simpson (1965); linear absorption coefficients from *International Tables for X-ray Crystallography* (1974).* Positional and

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å$^2$) *for non*-H *atoms of* $C_{19}H_{23}NO$

| | *x* | *y* | *z* | $U_{\rm eq}$ |
|---|---|---|---|---|
| C1 | 0·7286 (2) | 0·24979 (15) | 0·05774 (11) | 0·0206 (4) |
| C2 | 0·8388 (2) | 0·31265 (14) | 0·11597 (11) | 0·0200 (4) |
| N3 | 0·88565 (14) | 0·23574 (12) | 0·18211 (9) | 0·0185 (3) |
| C4 | 0·8254 (2) | 0·11311 (14) | 0·17696 (10) | 0·0199 (4) |
| C5 | 0·7702 (2) | 0·0698 (2) | 0·26898 (11) | 0·0265 (5) |
| C6 | 0·6427 (2) | 0·0252 (2) | 0·28122 (11) | 0·0304 (5) |
| C7 | 0·5353 (2) | 0·0174 (2) | 0·20568 (12) | 0·0294 (5) |
| C8 | 0·5615 (2) | 0·10996 (15) | 0·12866 (11) | 0·0220 (4) |
| C9 | 0·5211 (2) | 0·2402 (2) | 0·15223 (13) | 0·0280 (5) |
| C10 | 0·5847 (2) | 0·3103 (2) | 0·07084 (13) | 0·0292 (5) |
| C11 | 0·7168 (2) | 0·11882 (14) | 0·09593 (10) | 0·0189 (4) |
| C12 | 0·7483 (2) | 0·0212 (2) | 0·02435 (13) | 0·0321 (6) |
| O13 | 0·87811 (14) | 0·41774 (11) | 0·10568 (9) | 0·0306 (4) |
| C14 | 1·0024 (2) | 0·2694 (2) | 0·24361 (11) | 0·0203 (4) |
| C15 | 1·1224 (2) | 0·1786 (2) | 0·23638 (13) | 0·0287 (5) |
| C16 | 0·9535 (2) | 0·29572 (14) | 0·34256 (11) | 0·0195 (4) |
| C17 | 1·0203 (2) | 0·2477 (2) | 0·41995 (12) | 0·0263 (5) |
| C18 | 0·9784 (2) | 0·2805 (2) | 0·50976 (12) | 0·0314 (5) |
| C19 | 0·8691 (2) | 0·3602 (2) | 0·52198 (12) | 0·0307 (5) |
| C20 | 0·7996 (2) | 0·4073 (2) | 0·44549 (14) | 0·0316 (5) |
| C21 | 0·8417 (2) | 0·3751 (2) | 0·35622 (12) | 0·0251 (4) |

$U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^* A_{ij}$ where $A_{ij}$ is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

Table 2. *Bond lengths* (Å) *and angles* (°) *for non*-H *atoms of* $C_{19}H_{23}NO$

| 1 | 2 | 3 | 1–2 | 1–2–3 |
|---|---|---|---|---|
| C2 | C1 | C10 | 1·512 (2) | 110·63 (14) |
| C10 | C1 | C11 | 1·538 (2) | 107·29 (13) |
| C11 | C1 | C2 | 1·553 (2) | 106·47 (13) |
| N3 | C2 | O13 | 1·351 (2) | 125·3 (2) |
| N3 | C2 | C1 | | 109·25 (13) |
| O13 | C2 | C1 | 1·230 (2) | 125·40 (15) |
| C4 | N3 | C14 | 1·474 (2) | 123·94 (12) |
| C4 | N3 | C2 | | 114·50 (13) |
| C14 | N3 | C2 | 1·469 (2) | 120·93 (13) |
| C5 | C4 | C11 | 1·502 (2) | 115·94 (14) |
| C5 | C4 | N3 | | 112·67 (13) |
| C11 | C4 | N3 | 1·560 (2) | 105·01 (12) |
| C6 | C5 | C4 | 1·324 (3) | 123·9 (2) |
| C7 | C6 | C5 | 1·495 (3) | 123·6 (2) |
| C8 | C7 | C6 | 1·528 (2) | 112·1 (2) |
| C9 | C8 | C11 | 1·529 (3) | 104·31 (13) |
| C9 | C8 | C7 | | 115·41 (14) |
| C11 | C8 | C7 | 1·557 (2) | 114·62 (14) |
| C10 | C9 | C8 | 1·528 (3) | 102·00 (14) |
| C1 | C10 | C9 | | 103·14 (14) |
| C12 | C11 | C1 | 1·521 (2) | 114·10 (13) |
| C12 | C11 | C4 | | 110·18 (14) |
| C12 | C11 | C8 | | 110·34 (14) |
| C1 | C11 | C4 | | 104·67 (12) |
| C1 | C11 | C8 | | 103·57 (13) |
| C4 | C11 | C8 | | 113·80 (12) |
| C15 | C14 | C16 | 1·526 (3) | 114·72 (14) |
| C15 | C14 | N3 | | 111·11 (14) |
| C16 | C14 | N3 | 1·525 (2) | 112·20 (13) |
| C17 | C16 | C21 | 1·387 (2) | 118·62 (15) |
| C17 | C16 | C14 | | 122·31 (14) |
| C21 | C16 | C14 | 1·395 (2) | 119·01 (14) |
| C18 | C17 | C16 | 1·399 (2) | 120·6 (2) |
| C19 | C18 | C17 | 1·375 (3) | 120·0 (2) |
| C20 | C19 | C18 | 1·385 (3) | 120·1 (2) |
| C21 | C20 | C19 | 1·390 (3) | 119·9 (2) |
| C16 | C21 | C20 | | 120·8 (2) |

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* Lists of anisotropic thermal parameters, positional and isotropic thermal parameters for the H atoms, bond lengths 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 44312 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

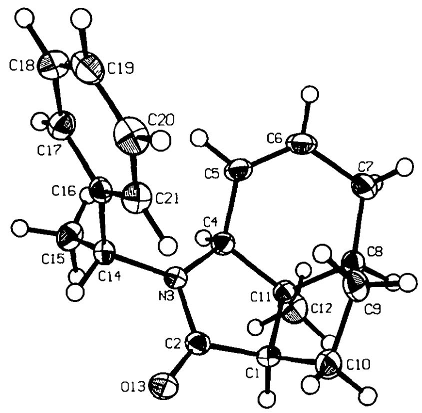

Fig. 1. View showing atom-labelling scheme. Thermal ellipsoids are scaled to the 50% probability level.

thermal parameters for non-H atoms are listed in Table 1, while bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labelling scheme is shown in Fig. 1. The least-squares-planes program was supplied by Cordes (1983); other computer programs from reference 11 of Gadol & Davis (1982).

**Related literature.** (1) is a synthetic precursor to the naturally occurring alkaloid dendrobine. The tricyclic ring system of (1) with various functional groups has been used as an intermediate in the total synthesis of dendrobine (Inubushi, Kikuchi, Ibuka, Tanaka, Saji & Tokane, 1972; Yamada, Suzuki, Hayakawa, Aoki, Nakamura, Nagase & Hirata, 1972; Kende, Bentley, Mader & Ridge, 1974; Roush, 1980).

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# 2-(1,2-Dibromoethyl)-1,4-benzodioxan

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**Abstract.** $C_{10}H_{10}Br_2O_2$, $M_r = 322{\cdot}00$, monoclinic, $P2_1/c$, $a = 9{\cdot}4088\ (9)$, $b = 19{\cdot}616\ (2)$, $c = 5{\cdot}958\ (1)$ Å, $\beta = 94{\cdot}61\ (2)°$, $V = 1095{\cdot}99$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}95$ Mg m$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 7{\cdot}21$ mm$^{-1}$, $F(000) = 624$, $T = 293$ K, $R = 0{\cdot}036$ for 1357 reflexions. The crystals examined proved to be the racemate of the *R,S* isomer. The dioxan ring adopts a half-chair conformation with the ethyl group *trans* to the dioxan C—C bond. The Br atoms are *trans* to each other, with the torsion angle Br—C—C—Br = 170·2 (4)°.

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