

$S = 0.772$   
 2006 reflections  
 217 parameters  
 H-atom parameters not  
 refined  
 $w = 1/[\sigma^2(F) + 0.0095F^2]$   
 $(\Delta/\sigma)_{\max} = 0.005$

Atomic scattering factors  
 from *International Tables*  
 for *X-ray Crystallography*  
 (1974, Vol. IV, Table  
 2.3.1) for C, N, O and  
 S, and Stewart, Davidson  
 & Simpson (1965) for H

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: TA1011). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
S	0.37386 (4)	0.00710 (7)	0.19207 (6)	0.0443 (3)
N	0.2950 (1)	-0.0512 (2)	0.2238 (2)	0.035 (1)
O(1)	0.3518 (1)	-0.2064 (3)	0.2838 (2)	0.059 (1)
O(2)	0.2355 (1)	-0.2043 (2)	0.2931 (2)	0.055 (1)
O(3)	0.3528 (1)	0.1101 (2)	0.1481 (2)	0.061 (1)
O(4)	0.4195 (1)	0.0163 (3)	0.2636 (2)	0.066 (1)
C(1)	0.2971 (2)	-0.1558 (3)	0.2675 (2)	0.041 (1)
C(2)	0.1668 (2)	-0.1510 (3)	0.2750 (2)	0.050 (1)
C(3)	0.1701 (2)	-0.0225 (3)	0.2612 (2)	0.040 (1)
C(4)	0.2270 (2)	0.0050 (3)	0.1954 (2)	0.034 (1)
C(5)	0.0965 (2)	0.0246 (3)	0.2379 (3)	0.052 (1)
C(6)	0.2093 (2)	-0.0301 (3)	0.1047 (2)	0.034 (1)
C(7)	0.2204 (2)	-0.1420 (3)	0.0742 (2)	0.044 (1)
C(8)	0.2058 (2)	-0.1705 (3)	-0.0090 (2)	0.053 (1)
C(9)	0.1793 (2)	-0.0877 (4)	-0.0626 (2)	0.056 (1)
C(10)	0.1667 (2)	0.0247 (4)	-0.0343 (2)	0.058 (1)
C(11)	0.1821 (2)	0.0539 (3)	0.0498 (2)	0.046 (1)
C(12)	0.4114 (2)	-0.0896 (3)	0.1183 (2)	0.041 (1)
C(13)	0.4603 (2)	-0.1740 (3)	0.1440 (2)	0.046 (1)
C(14)	0.4911 (2)	-0.2460 (3)	0.0842 (3)	0.051 (1)
C(15)	0.4747 (2)	-0.2360 (3)	-0.0018 (2)	0.052 (1)
C(16)	0.4246 (2)	-0.1519 (4)	-0.0252 (2)	0.063 (1)
C(17)	0.3932 (2)	-0.0782 (4)	0.0328 (2)	0.058 (1)
C(18)	0.5092 (2)	-0.3132 (4)	-0.0673 (3)	0.073 (2)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

S—N	1.694 (3)	O(2)—C(1)	1.339 (4)
S—O(3)	1.422 (3)	O(2)—C(2)	1.451 (4)
S—O(4)	1.420 (3)	C(2)—C(3)	1.485 (5)
S—C(12)	1.753 (3)	C(3)—C(4)	1.521 (4)
N—C(1)	1.381 (4)	C(3)—C(5)	1.521 (5)
N—C(4)	1.493 (4)	C(4)—C(6)	1.523 (4)
O(1)—C(1)	1.204 (4)		
N—S—O(3)	103.3 (1)	O(1)—C(1)—O(2)	117.8 (3)
N—S—O(4)	108.5 (1)	O(2)—C(2)—C(3)	114.1 (3)
O(3)—S—O(4)	119.6 (2)	C(2)—C(3)—C(4)	109.5 (3)
N—S—C(12)	107.3 (1)	C(2)—C(3)—C(5)	110.4 (3)
O(3)—S—C(12)	108.0 (2)	C(4)—C(3)—C(5)	113.2 (3)
O(4)—S—C(12)	109.6 (2)	N—C(4)—C(3)	107.5 (2)
S—N—C(1)	117.7 (2)	N—C(4)—C(6)	110.8 (2)
S—N—C(4)	118.8 (2)	C(3)—C(4)—C(6)	115.9 (3)
C(1)—N—C(4)	123.2 (2)	C(4)—C(6)—C(7)	122.6 (3)
C(1)—O(2)—C(2)	121.8 (3)	C(4)—C(6)—C(11)	119.0 (3)
N—C(1)—O(1)	123.1 (3)	S—C(12)—C(13)	120.5 (2)
N—C(1)—O(2)	119.0 (3)	S—C(12)—C(17)	119.0 (3)

The scan width was  $\pm(0.75 + 0.2\tan\theta)^\circ$  in  $\theta$  from the calculated Bragg angle. Measurements were made using a scan speed of  $0.04^\circ \text{ s}^{-1}$  and background counts were collected for 50% of the scan time on each side of every scan. Data collection, cell refinement and data reduction: Philips PW1100 software. Structure solution and refinement: *SHELX76* (Sheldrick, 1976). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990).

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## (±)-[6 $\alpha$ (H)]-7,7,9 $\beta$ -Trimethyl-cis-tricyclo[6.3.1.0<sup>1,6</sup>]dodecan-2-one

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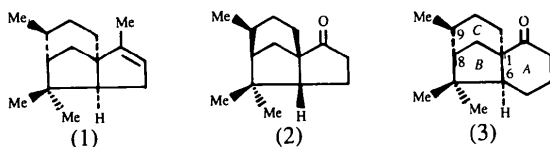
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## Abstract

The title compound, C<sub>15</sub>H<sub>24</sub>O, possesses a bridged tricyclic structure with the cyclohexanone ring (A) and the cyclopentane ring (B) *cis* fused. The conformations of rings A and B can be best described as a distorted boat and an envelope, respectively. The cyclohexane ring (C) adopts a distorted chair conformation and the secondary methyl group at C9 is *cis* to the H atom at C8 [C13—C9—C8—H8 -52.6(3) $^\circ$ ].

**Comment**

$\Delta^2$ -Cedrene, (1), a tricyclic sesquiterpene containing a *cis*-tricyclo[5.3.1.0<sup>1,5</sup>]undecane skeleton, was isolated from vetiver oil (Kaiser & Naegeli, 1972). The synthesis and structural characterization of the C8 epimer of  $\Delta^2$ -cedrene using the ketone (2) as a key intermediate has been reported recently (Chen & Lin, 1992). In connection with our studies of the synthesis of bridged ring systems related to tricyclic sesquiterpenes (Das, Karpha, Ghosal & Mukherjee, 1992; Ghosal, Saha & Mukherjee, 1990), the tricyclic ketone (3) has been prepared *via* the aryl-participated intramolecular cyclization of an appropriately substituted indan derivative, and has been purified and crystallized. This ketone possesses the requisite structural features for a potential intermediate in a total synthesis of  $\Delta^2$ -cedrene. The determination of the structure of the title compound (3) was undertaken in order to establish the relative stereochemistries of the four asymmetric centres (C1, C6, C8 and C9) and to plan the subsequent sequence of reactions leading to the total synthesis of (1).



The *ORTEPII* (Johnson, 1976) view of the title compound given in Fig. 1 shows that the molecule consists of a bicyclic bridged system (rings B and C) *cis* fused with the cyclohexanone ring A [torsion angle C11—C1—C6—H6  $-22(3)^\circ$ ]. The results of the present structural analysis, in which the C—C bond lengths range from 1.508(7) to 1.569(5) Å, are in agreement with the results of the determination of the structure of  $\beta$ -homopipizolone (Huipe-Nava *et al.*, 1993). The H atom at C8 and the methyl group at C9 display *cis* geometry [torsion angle C13—C9—C8—H8  $-52.6(3)^\circ$ ]. The cyclohexanone ring A adopts a distorted boat conformation with C2 and C5 displaced by 0.380(3) and 0.716(5) Å, respectively, in the same direction from the basal plane through C1, C3, C4 and C6. The cyclohexane ring C has a distorted chair conformation with C12 0.888(4) Å above and C10 0.504(4) Å below the least-squares plane defined by the remaining endocyclic atoms. The cyclopentane ring B has an envelope conformation with C12 occupying the flap position and the C14 and C15 methyl groups in equatorial and axial orientations, respectively. The dihedral angle between the planar parts of rings A and B is  $15.4(2)^\circ$ . The Cremer & Pople (1975) ring-puckering parameters are  $q_2 = 0.640(4)$ ,  $q_3 = -0.135(4)$ ,  $Q = 0.654(4)$ ,  $\theta = 101.9(3)^\circ$  and  $\varphi = 62.7(4)^\circ$  for ring A,  $q_2 = 0.483(3)$  and  $\varphi = -49.5(4)^\circ$  for ring B, and  $q_2 =$

$0.261(4)$ ,  $q_3 = 0.603(3)$ ,  $Q = 0.657(3)$ ,  $\theta = 23.4(3)^\circ$  and  $\varphi = 51.7(7)^\circ$  for ring C. The molecular packing is determined by van der Waals contacts only.

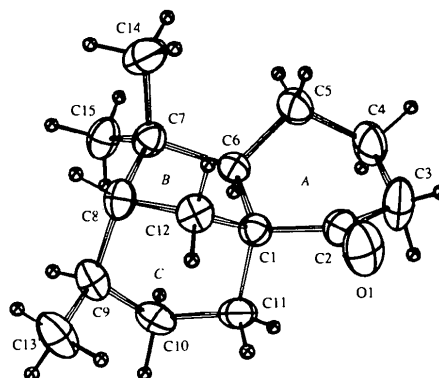


Fig. 1. An *ORTEPII* (Johnson, 1976) view of the molecule showing the atom-labelling and ring-numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

**Experimental***Crystal data*

C<sub>15</sub>H<sub>24</sub>O  
*M<sub>r</sub>* = 220.34  
 Orthorhombic  
*P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 9.958(3) Å  
*b* = 9.961(4) Å  
*c* = 12.978(6) Å  
*V* = 1287.3(9) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.137 Mg m<sup>-3</sup>

Cu K $\alpha$  radiation  
 $\lambda$  = 1.5418 Å  
 Cell parameters from 20 reflections  
 $\theta$  = 10–20°  
 $\mu$  = 0.518 mm<sup>-1</sup>  
*T* = 294(2) K  
 Block  
 0.5 × 0.5 × 0.25 mm  
 Colourless

*Data collection*

AFC-5 diffractometer  
 2 $\theta$ – $\omega$  scans  
 Absorption correction: empirical  
 $T_{\min} = 0.895$ ,  $T_{\max} = 1.000$   
 1111 measured reflections  
 1111 independent reflections  
 1049 observed reflections  
 [ $I > 2\sigma(I)$ ]

$\theta_{\max} = 59.94^\circ$   
 $h = 0 \rightarrow 11$   
 $k = 0 \rightarrow 11$   
 $l = 0 \rightarrow 14$   
 3 standard reflections monitored every 150 reflections  
 intensity decay: 4.8%

*Refinement*

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.0502$   
 $wR(F^2) = 0.1217$   
 $S = 0.841$   
 1111 reflections  
 241 parameters  
 $w = 1/[\sigma^2(F_o^2) + (0.1095P)^2 + 0.4469P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.19$   
 $\Delta\rho_{\max} = 0.16 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}$
O1	0.0535 (3)	0.1891 (3)	-0.2387 (2)	0.0756 (9)
C1	0.0728 (3)	0.0358 (3)	-0.0994 (2)	0.0390 (7)
C2	0.0021 (4)	0.0987 (3)	-0.1903 (2)	0.0491 (8)
C3	-0.1324 (5)	0.0417 (6)	-0.2210 (3)	0.0758 (13)
C4	-0.2199 (4)	-0.0062 (6)	-0.1333 (3)	0.0701 (12)
C5	-0.1646 (4)	0.0368 (5)	-0.0290 (3)	0.0598 (10)
C6	-0.0236 (3)	-0.0186 (3)	-0.0147 (2)	0.0390 (7)
C7	0.0481 (3)	0.0110 (3)	0.0905 (2)	0.0446 (8)
C8	0.1922 (3)	0.0537 (3)	0.0566 (2)	0.0449 (8)
C9	0.2890 (3)	-0.0637 (4)	0.0318 (3)	0.0526 (9)
C10	0.2338 (4)	-0.1533 (3)	-0.0541 (3)	0.0547 (9)
C11	0.1640 (3)	-0.0781 (4)	-0.1413 (3)	0.0497 (8)
C12	0.1641 (4)	0.1329 (3)	-0.0415 (3)	0.0452 (8)
C13	0.4296 (4)	-0.0111 (6)	0.0071 (5)	0.0752 (13)
C14	-0.0143 (5)	0.1283 (4)	0.1492 (3)	0.0671 (11)
C15	0.0432 (4)	-0.1097 (4)	0.1624 (3)	0.0590 (10)

Table 2. Selected geometric parameters (Å, °)

O1—C2	1.210 (5)	C6—C7	1.569 (5)
C1—C2	1.509 (5)	C7—C15	1.522 (6)
C1—C12	1.526 (6)	C7—C14	1.528 (7)
C1—C6	1.557 (6)	C7—C8	1.560 (6)
C1—C11	1.552 (6)	C8—C12	1.524 (6)
C2—C3	1.508 (7)	C8—C9	1.549 (6)
C3—C4	1.511 (8)	C9—C13	1.529 (7)
C4—C5	1.522 (8)	C9—C10	1.530 (7)
C5—C6	1.520 (6)	C10—C11	1.525 (6)
C2—C1—C6	114.1 (3)	C1—C6—C7	105.6 (3)
C12—C1—C6	103.9 (3)	C15—C7—C14	106.6 (4)
C12—C1—C11	106.7 (3)	C8—C7—C6	103.0 (3)
O1—C2—C1	121.1 (4)	C12—C8—C9	109.4 (4)
O1—C2—C3	121.2 (4)	C12—C8—C7	102.0 (3)
C1—C2—C3	117.6 (4)	C10—C9—C8	111.6 (4)
C4—C3—C2	115.6 (4)	C11—C10—C9	114.7 (4)
C3—C4—C5	111.9 (4)	C10—C11—C1	111.4 (3)
C4—C5—C6	109.9 (4)	C8—C12—C1	101.1 (3)
C5—C6—C1	110.9 (3)		

Symmetry-related reflections for the tetragonal system were checked but were not found to be equivalent, so orthorhombic symmetry was assumed. The similarity of the unit-cell parameters *a* and *b* was concluded to be fortuitous. The structure was solved by direct methods (*MULTAN88*; Debaerdemaeker *et al.*, 1988) followed by Fourier methods and refined by anisotropic full-matrix least-squares on  $F^2$  using *SHELXL93* (Sheldrick, 1993). H atoms were located from difference Fourier maps and were refined isotropically. Data collection: AFC-5 software. Software used for geometric calculations: *PARST* (Nardelli, 1983). Software used to prepare material for publication: *SHELXL93*. Molecular graphics: *ORTEPII* (Johnson, 1976). Calculations were performed using a VAX3400 computer at the Computer Center, Indian Association for the Cultivation of Science.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: LI1123). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Quitenine Ethyl Ester

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## Abstract

The crystal structure of 6'-methoxy-(8 $\alpha$ ,9*R*)-cinchonan-9-ol-3-carboxylic acid ethyl ester, C<sub>21</sub>H<sub>26</sub>N<sub>2</sub>O<sub>4</sub>, has been determined by X-ray diffraction. The conformation of the molecule is closed, in contrast to the open conformation of quinine. The N atom of the quinuclidine moiety is not involved in hydrogen bonding. The molecules linked by intermolecular N13 $\cdots$ H—O22 hydrogen bonds form chains along the *y* axis.

## Comment

Quitenine ethyl ester, (II), is a derivative of the anti-malarial alkaloid quinine, (I), in which the vinyl group at C3 of the quinuclidine moiety is oxidized to carboxyl

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