ORTEP (Johnson, 1965) drawing of the molecule with atomic labelling. Fig. 2 shows the crystal structure. Fig. 1 clearly indicates the (*E*,*Z*)-conformation. The internal rotation angles of N(1)—N(2)—N(3)—C(6) and N(3)—N(2)—N(1)—C(4) are 2·8 (2) and − 174·6 (2)°, respectively.

The N(1)—N(2), N(2)—N(3) distances, 1·296 (2), 1·299 (2) Å respectively, indicate remarkable electron delocalization. A more detailed experiment carried out for (1*E*,2*Z*)- and (1*Z*,2*E*)-2,3-dimethyl-1-phthalimidoazimine (Moor, Grieb, Niggli, Hoesch & Dreiding, 1979) indicates 1·301 (10), 1·332 (6) Å for N(1)—N(2) and 1·268 (9), 1·263 (5) Å for N(2)—N(3) distances, respectively. The differences of 0·03 and 0·06 Å between the N(1)—N(2) and N(2)—N(3) distances, respectively, are caused by the steric and electronic effects of the phthalimido moiety connected to N(1). In the case of (I) the azimine moiety, having identical substituents connected to N(1) and N(2) whose bond lengths suggest the N═N character, exhibits a higher 1,3-dipolar contribution (Moor *et al.*, 1979). The phenyl ring is twisted about the N(2)—C(8) bond to reduce the steric hindrance between C(4) and H(9); the non-bonded separation is 2·7 (2) Å. The dihedral angle between the phenyl ring and the azimine plane is 58·1 (2)°.

In the crystal structure the molecules within a column are held together by the perpendicular stacking. Intermolecular hydrogen bonds and unusual short non-bonded contacts are not observed.

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Structure of 1-Hydroxy-10-isopropyl-7-methyl-*cis*-bicyclo[4.4.0]decan-3-one*

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Abstract. $C_{14}H_{24}O_2$, $M_r = 224·3$, monoclinic, $P2_1$, $a = 7·400$ (1), $b = 15·967$ (2), $c = 11·755$ (2) Å, $\beta = 105·79$ (1)°, $V = 1336·5$ (2) Å^3, $Z = 4$, D_m (flotation in KBr solution) = 1·12, $D_x = 1·11$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0·79$ cm^{-1}, $F(000) = 496$, $T = 293$ K, $R = 0·049$ for 1749 observed reflections. The structure has a non-steroid *cis* conformation with its decalin ring in double-chair form. The structure is stabilized by two intermolecular O—H···O hydrogen bonds [2·956 (4) and 2·933 (4) Å].

Introduction. The sesquiterpene lactone 'qinghaosu' or artimisinin is an antimalarial drug. In an attempt to synthesize totally this key product the title compound was synthesized (Salunke, Phadke & Kulkarni, 1988). X-ray structure determination was essential to establish uniquely the stereochemistry of this key intermediate.

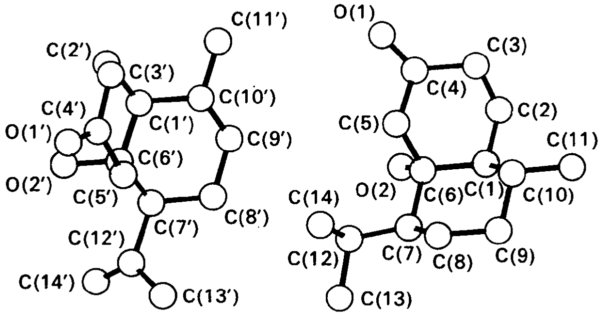

Fig. 1. *PLUTO* diagram (Motherwell & Clegg, 1978) of the molecule showing the atomic numbering scheme.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters for non-*H *atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
O(1)	3745 (4)	965	2803 (3)	5·08 (9)
O(2)	1190 (3)	2557 (2)	772 (2)	3·47 (6)
C(1)	−437 (5)	1349 (3)	−301 (3)	2·82 (8)
C(2)	1460 (5)	951 (3)	−275 (4)	3·36 (11)
C(3)	2295 (6)	465 (3)	872 (4)	3·97 (12)
C(4)	2378 (5)	973 (3)	1937 (4)	3·41 (11)
C(5)	624 (5)	1459 (3)	1939 (3)	3·17 (10)
C(6)	−196 (5)	1932 (2)	790 (3)	2·62 (8)
C(7)	−2094 (5)	2370 (2)	737 (3)	2·79 (8)
C(8)	−3693 (5)	1726 (3)	572 (4)	3·24 (10)
C(9)	−3870 (5)	1176 (3)	−498 (4)	3·69 (11)
C(10)	−2045 (5)	711 (3)	−415 (4)	3·30 (11)
C(11)	−2248 (6)	134 (4)	−1473 (5)	5·03 (13)
C(12)	−2033 (5)	2995 (3)	1741 (4)	3·44 (11)
C(13)	−3259 (8)	3755 (4)	1262 (5)	6·03 (17)
C(14)	−2566 (7)	2633 (4)	2811 (4)	5·06 (13)
O(1′)	286 (5)	3599 (3)	8612 (3)	5·80 (11)
O(2′)	2796 (4)	5111 (2)	7304 (2)	3·63 (6)
C(1′)	3883 (5)	3868 (3)	6491 (4)	3·57 (11)
C(2′)	4501 (6)	3496 (3)	7735 (4)	4·49 (12)
C(3′)	2896 (7)	3024 (3)	8061 (4)	4·64 (13)
C(4′)	1190 (6)	3558 (3)	7888 (4)	4·18 (12)
C(5′)	592 (5)	4045 (3)	6747 (3)	3·29 (10)
C(6′)	2219 (5)	4482 (3)	6413 (3)	3·10 (10)
C(7′)	1621 (6)	4898 (3)	5188 (4)	3·50 (11)
C(8′)	1238 (6)	4255 (3)	4195 (3)	4·26 (11)
C(9′)	2913 (7)	3679 (3)	4294 (4)	5·00 (13)
C(10′)	3465 (6)	3233 (3)	5478 (4)	4·23 (12)
C(11′)	5146 (8)	2640 (4)	5545 (6)	6·55 (19)
C(12′)	58 (8)	5567 (3)	5033 (4)	4·58 (11)
C(13′)	−1964 (8)	5251 (5)	4549 (5)	6·61 (18)
C(14′)	394 (9)	6295 (4)	4284 (5)	6·78 (19)

Experimental. Crystal approximately 0·45 × 0·25 × 1·00 mm, Enraf–Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta < 23{\cdot}5°$, h 0 to 8, k 0 to 17, l −14 to 14, 2240 reflections collected, 1749 judged significant ($|F_o| \geq 3\sigma|F_o|$), lattice parameters from 20 reflections ($22 < 2\theta < 42°$), three standard reflections ($25\bar{1}$, $\bar{3}63$, 273) every 3000 s, 4% variation in intensity, no correction for absorption, structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Least-squares refinement (*LALS*; Gantzel, Sparks & Trueblood, 1961) of scale factor, positional and anisotropic thermal parameters for non-H atoms (H positions from a difference Fourier map and isotropic thermal parameters from non-H atoms to which they are attached; all these parameters held fixed during refinement) converged to $R = 0{\cdot}049$, $wR = 0{\cdot}047$, $S = 0{\cdot}97$, $w(|F_o| - |F_c|)^2$ minimized, $w = (3{\cdot}0 + 1{\cdot}0|F_o| + 0{\cdot}02|F_o|^2)^{-1}$, $(\Delta/\sigma)_{\max} = 0{\cdot}1$, final $\Delta\rho$ excursions $< |0{\cdot}2|$ e Å^{-3}. No corrections for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

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

	Molecule (1)	Molecule (2)
O(1)—C(4)	1·224 (5)	1·218 (6)
O(2)—C(6)	1·436 (5)	1·430 (5)
C(1)—C(2)	1·533 (5)	1·528 (6)
C(1)—C(6)	1·556 (5)	1·557 (6)
C(1)—C(10)	1·544 (6)	1·530 (6)
C(2)—C(3)	1·533 (6)	1·540 (7)
C(3)—C(4)	1·478 (6)	1·491 (7)
C(4)—C(5)	1·513 (6)	1·509 (6)
C(5)—C(6)	1·522 (5)	1·532 (6)
C(6)—C(7)	1·555 (5)	1·538 (6)
C(7)—C(8)	1·539 (6)	1·521 (6)
C(7)—C(12)	1·537 (6)	1·549 (7)
C(8)—C(9)	1·510 (6)	1·523 (7)
C(9)—C(10)	1·520 (6)	1·517 (7)
C(10)—C(11)	1·522 (7)	1·549 (8)
C(12)—C(13)	1·528 (7)	1·535 (8)
C(12)—C(14)	1·530 (7)	1·518 (8)
C(2)—C(1)—C(6)	109·5 (3)	109·1 (3)
C(2)—C(1)—C(10)	114·1 (3)	115·6 (4)
C(6)—C(1)—C(10)	112·3 (3)	112·5 (3)
C(1)—C(2)—C(3)	112·0 (3)	112·2 (4)
C(2)—C(3)—C(4)	112·4 (4)	111·6 (4)
O(1)—C(4)—C(3)	122·2 (4)	122·7 (4)
O(1)—C(4)—C(5)	121·2 (4)	120·6 (4)
C(3)—C(4)—C(5)	116·4 (4)	116·7 (4)
C(4)—C(5)—C(6)	112·9 (3)	113·7 (3)
O(2)—C(6)—C(1)	109·0 (3)	109·0 (3)
O(2)—C(6)—C(5)	104·1 (3)	103·4 (3)
O(2)—C(6)—C(7)	109·2 (3)	109·5 (3)
C(1)—C(6)—C(5)	111·3 (3)	111·2 (3)
C(1)—C(6)—C(7)	109·6 (3)	110·4 (3)
C(5)—C(6)—C(7)	113·4 (3)	113·0 (3)
C(6)—C(7)—C(8)	111·1 (3)	111·9 (3)
C(6)—C(7)—C(12)	115·0 (3)	115·1 (4)
C(8)—C(7)—C(12)	113·1 (3)	113·5 (4)
C(7)—C(8)—C(9)	112·6 (3)	111·8 (4)
C(8)—C(9)—C(10)	110·8 (3)	111·3 (4)
C(1)—C(10)—C(9)	109·5 (3)	110·5 (4)
C(1)—C(10)—C(11)	111·8 (4)	112·1 (4)
C(9)—C(10)—C(11)	110·8 (4)	110·2 (4)
C(7)—C(12)—C(13)	110·0 (4)	116·0 (4)
C(7)—C(12)—C(14)	115·2 (4)	111·2 (4)
C(13)—C(12)—C(14)	110·1 (4)	109·5 (5)

Discussion. Fig. 1 gives a perspective view of the molecule with atomic numbering. The atomic parameters along with their e.s.d.'s and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1* while Table 2 gives the bond lengths and angles.

There are two molecules per asymmetric unit. The relation between the two molecules is x, y, z; x', y', z' where $y - y' \simeq 0{\cdot}26$ which rules out the possibility of any other space group with only one molecule in the asymmetric unit. The structure belongs to the *cis*-decalin class of compounds and adopts a non-steroid conformation with its two six-membered rings in 'double-chair' conformation. The hydroxy group is axial and the hydrogen on C(1) is equatorial to the cyclohexanone moiety. The bond length [C(1)—C(6)] which fuses the two six-membered rings is 1·556 (5) and 1·557 (6) Å which is significantly longer than the standard Csp^3—Csp^3 bond length, 1·533 (3) Å (Bartell, 1959). Table 3 lists some similar molecules which show such lengthening of the central C—C

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*Lists of structure factors, anisotropic thermal parameters for non-H atoms, and coordinates and isotropic thermal parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51956 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Some similar molecules with elongation of the bond at the fusion of the two rings*

Compound	C(1)—C(6)	Reference
3-Hydroxy-*trans*-bicyclo[4.4.0]decane-1,3-carbolactone	1·562 (5) Å	*a*
3-Methoxy-*trans*-bicyclo[4.4.0]decane-1,3-carbolactone	1·558 (5)	*a*
Methyl 3-oxo-*trans*-bicyclo[4.4.0]decane-1-carboxylate	1·558 (3)	*a*
3-Oxo-*cis*-bicyclo[4.4.0]decane-1-carboxylic acid	1·557 (3)	*a*
1-Carboxy-*trans*-bicyclo[4.4.0]decan-3-one ethylene acetal	1·556 (8)	*b*
3-Amino-3-hydroxy-*trans*-bicyclo[4.4.0]-decane-1-carboxylic acid lactam	1·553 (5)	*c*
cis-Perfluorobicyclo[4.4.0]decane-1,6-diol	1·581 (8)	*d*
5-Deoxy-5-hydroperoxy-5-epitelekin	1·557 (4)	*e*
O-Methylbaccharocephol	1·561 (9)	*f*
Present study	1·556 (5) 1·557 (6)	

References: (*a*) Chadwick & Dunitz (1979). (*b*) Chadwick & Dunitz (1978*a*). (*c*) Chadwick & Dunitz (1978*b*). (*d*) Hamor & Hamor (1976). (*e*) Appendino *et al.* (1984). (*f*) Rivera, Castillo & Rodriguez (1988).

bond. The two rings are folded as seen from Fig. 1 as well as from the torsion angles [C(5)—C(6)—C(1)—C(10) = 71·4 (4), 73·3 (3); C(3)—C(2)—C(1)—C(10) = −69·7 (4), −69·0 (5); C(5)—C(6)—C(7)—C(8) = −72·7 (4), −72·8 (4)°] and the non-bonded contacts [C(5)–C(10) = 3·164 (6), 3·186 (6); C(3)–C(10) = 3·180 (6), 3·195 (7); C(5)–C(8) = 3·190 (5), 3·181 (6) Å]. These values are similar in both molecules and are comparable with those of the decalin moiety in 5-epitelekin (Appendino, Calleri, Chiari & Viterbo, 1984).

The molecules in the unit cell are held together by two intermolecular hydrogen bonds [O(1′)···O(2)(x, y, $1+z$) = 2·956 (4); O(1)···O(2′)($1-x$, $y-\frac{1}{2}$, $1-z$) = 2·933 (4) Å].

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Structure de la Diacétylantioquine

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(*Reçu le* 15 *février* 1989, *accepté le* 20 *décembre* 1989)

Abstract. 12′,20-Di-*O*-acetylrodiasine, $C_{41}H_{44}N_2O_8.\frac{9}{2}H_2O$, $M_r = 773·89$, orthorhombic, $P2_12_12_1$, $a = 11·138$ (3), $b = 14·461$ (3), $c = 26·464$ (5) Å, $V = 4262·5$ Å^3, $Z = 4$, $D_x = 1·206$ g cm^{-3} (including water solvant), λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6·56$ cm^{-1}, $F(000) = 1652$, $T = 291$ K, $R = 0·086$ for 2297 observed reflections with $I > 2·5\sigma(I)$. Antioquine is a new alkaloid extracted from *Pseudoxandra sclerocarpa*. The absolute configuration (1*S*,1′*R*) has been deduced from that of its diacetyl derivative. The molecule belongs to the small group of the bi(benzyltetrahydroisoquinoline) alkaloids with a biphenyl bond. In the crystal, the molecules are packed by means of an important network of hydrogen bonds including a large number of water molecules.

Introduction. L'antioquine (1) est l'alcaloïde majoritaire extrait des écorces d'une Annonacée, *Pseudoxandra sclerocarpa*, récoltée en Colombie.

Sa structure plane a été complètement élucidée par dégradation chimique et analyse des données spectrales, études spectroscopiques de RMN à haut champ. Il s'agit d'une bi(benzyltétrahydroisoquinoléine) à liaison biphénylique (Cortes, Saez, Hocquemiller, Cavé & Cavé, 1985).

Pour déterminer la stéréochimie des centres d'asymétrie C1 et C1′, il a été fait appel au dichroïsme circulaire. L'existence d'une terminaison ascendante dans la courbe de DC entre 225 et 210 nm (Leet, Elango, Hussain & Shamma, 1983) a permis d'établir la configuration (1*S*), identique à

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