dihedral angles. The dimensions and conformation of the norbornane ring system agree well with those of other substituted camphors (Allen & Rogers, 1971; Bear & Trotter, 1975; Baert & Fouret, 1978; Couldwell, Prout, Robey, Taylor & Rossotti, 1978; Phillips & Trotter, 1977; Rendle & Trotter, 1975). The molecular packing is illustrated in Fig. 2. The molecules are linked together by intermolecular N—H···O hydrogen bonds of 2·82 Å.

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Structure and Conformation of Incensole Oxide

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Abstract. $C_{20}H_{34}O_3$, orthorhombic, $P2_12_12_1$, a = 18·900 (9), b = 15·607 (7), c = 6·476 (3) Å, Z = 4, D_c = 1·12 Mg m^{-3}, Cu $K\alpha$ radiation, μ(Cu $K\alpha$) = 58·1 mm^{-1}. R = 0·057 and R_w = 0·076 for 1386 observed data with $I > 2\sigma(I)$. The structure is characterized by an oxirane ring and ether bridge. Their O atoms protrude outside and inside the diterpene macrocycle respectively. The double-bond C atoms and their substituents lie in a plane approximately perpendicular to the mean plane passing through the macrocycle atoms. Hydrogen bonds between the hydroxyl and epoxide O atoms of two different molecules occur along b. Strong van der Waals interactions stabilize the crystal structure along a and c.

Introduction. 11-Isopropyl-4,8,14-trimethyl-8,9;11,14-diepoxy-4-cyclotetradecen-1-ol* is a new macrocyclic diterpene, called incensole oxide (Nicoletti & Forcellese, 1968), isolated from frankincense, the resin produced by *Boswellia carteri*, together with incensole (Corsano & Nicoletti, 1967). This compound has been characterized by spectroscopic methods and chemical reactions by Nicoletti & Forcellese (1968). However, its conformation is unknown and some doubt exists about the position of the hydroxyl group. To establish the molecular geometry and conformation both of incensole and incensole oxide, the crystal structure of the latter was investigated. Since these compounds give rise to intriguing reactions, knowledge of their molecular structure can assist in interpreting the mechanisms.

Incensole oxide was crystallized at room temperature from an ether/n-hexane mixture by evaporation. Colourless flakes, elongated along b, m.p. 438 K, were obtained. A specimen with a maximum dimension about 1 mm was chosen to measure the cell constants and intensities within the range $4 \leq 2\theta \leq 120°$. Intensities were collected with Cu $K\alpha$ radiation (λ = 1·5418 Å) on an automatic four-circle Philips PW 1100 diffractometer equipped with a scintillation counter, pulse-height analyser and graphite monochromator. The $\omega/2\theta$ scanning mode was employed at a rate of 12° min^{-1} over a range of 2°. Background counts were taken for a constant time of 4 s. Three standard reflections, monitored every 4 h, remained

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* Note that the numbering used throughout this paper is arbitrary and does not correspond to that used in the systematic chemical name.

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essentially constant, showing only the deviations from the mean predicted by counting statistics. Lorentz and polarization corrections were applied taking into account the monochromator crystal (Azaroff, 1955) but no absorption corrections were made.

To solve the structure it was decided to resort simultaneously both to *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and to minimum-residual analysis (Damiani, Giglio, Liquori & Ripamonti, 1967). At first a molecular model useful for both the methods was developed, assuming standard bond lengths and angles, with a minimization program that tries to obtain closure in a chain or between two chains as a function of the internal rotation angles around the skeletal bonds (Conte, D'Ilario, Pavel & Giglio, 1979). The search of the minimum residual accomplished with this model was carried out in regions of the parametric space lacking short intermolecular contacts and favourable for the formation of hydrogen bonding between O(17) and O(20) belonging to the hydroxyl and epoxide groups of two different molecules (Fig. 1). Some packing arrangements, characterized by plausible hydrogen bonds joining rows of molecules parallel to *b*, which is the direction of maximum growth of the crystal, were found. Many of these molecular packings gave an unsatisfactory *R*, and, therefore, were discarded. Only one provided a promising *R*; this was used to calculate the phases of the structure factors.

At the same time the *E* map computed from the best set of phases obtained with *MULTAN* was interpreted with the aid of the molecular model. At this point it was clear that the best packing identified in the minimum-residual analysis agreed moderately well with the actual one. The slight disagreement was mainly due to the wrong position assigned to the hydroxyl group in

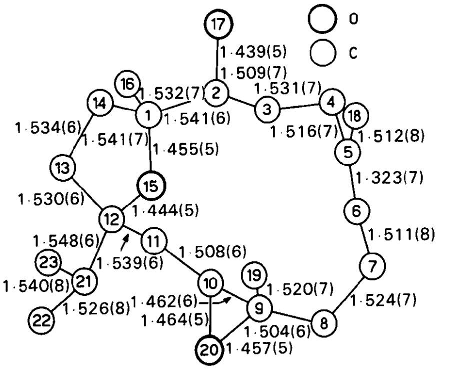

Fig. 1. Bond distances (Å), with e.s.d.'s in parentheses, of the incensole oxide molecule projected on the *ab* plane.

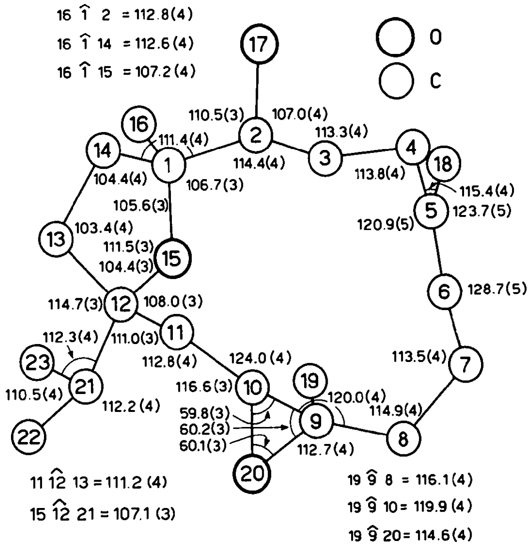

Fig. 2. Bond angles (°), with e.s.d.'s in parentheses, of the incensole oxide molecule projected on the *ab* plane.

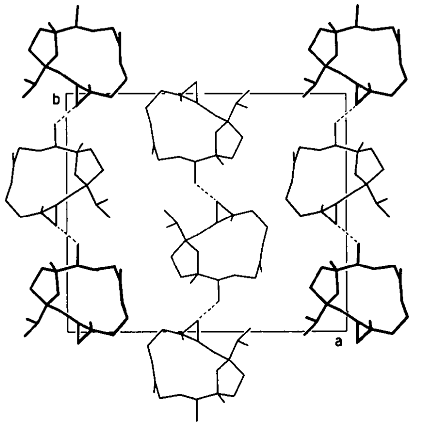

Fig. 3. Crystal packing of incensole oxide viewed along *c*. Broken lines represent hydrogen bonding.

Table 1. *Final fractional coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (*Hamilton,* 1959) *of the non-hydrogen atoms*

	x	*y*	*z*	*B* (Å^2)
C(1)	−358 (2)	2537 (3)	6177 (7)	3·2
C(2)	386 (2)	2831 (3)	5511 (7)	3·3
C(3)	965 (3)	2622 (3)	7035 (8)	3·9
C(4)	1710 (3)	2734 (3)	6147 (9)	4·5
C(5)	1878 (2)	2125 (3)	4390 (8)	3·9
C(6)	1979 (3)	1299 (3)	4738 (8)	4·3
C(7)	2154 (3)	598 (3)	3211 (9)	4·3
C(8)	1627 (2)	−142 (3)	3235 (8)	4·1
C(9)	904 (2)	74 (3)	2410 (7)	3·1
C(10)	351 (2)	372 (3)	3813 (7)	3·0
C(11)	−275 (2)	906 (3)	3149 (7)	3·1
C(12)	−752 (2)	1167 (3)	4968 (7)	2·8
C(13)	−1304 (2)	1828 (3)	4298 (7)	3·6
C(14)	−905 (3)	2682 (3)	4448 (7)	3·7
O(15)	−321 (1)	1613 (2)	6461 (4)	2·9
C(16)	−590 (3)	2925 (3)	8242 (8)	5·0
O(17)	399 (2)	3744 (2)	5204 (6)	4·8
C(18)	1914 (4)	2529 (4)	2271 (10)	6·0
C(19)	866 (3)	295 (3)	127 (7)	3·9
O(20)	361 (2)	−520 (2)	3117 (5)	3·6
C(21)	−1060 (2)	368 (3)	6063 (8)	3·7
C(22)	−1534 (4)	−158 (4)	4639 (11)	6·1
C(23)	−1455 (3)	598 (4)	8066 (10)	5·1

Table 2. *Some relevant internal torsion angles* (°) *of incensole oxide with e.s.d.'s in parentheses*

C(13)–C(14)–C(1)–C(2)	131·7 (4)	C(14)–C(1)–C(2)–C(3)	−173·7 (4)	C(1)–C(2)–C(3)–C(4)	167·0 (4)
C(2)–C(3)–C(4)–C(5)	−64·5 (5)	C(3)–C(4)–C(5)–C(6)	−71·5 (6)	C(4)–C(5)–C(6)–C(7)	−179·6 (5)
C(5)–C(6)–C(7)–C(8)	−123·6 (6)	C(6)–C(7)–C(8)–C(9)	69·2 (6)	C(7)–C(8)–C(9)–C(10)	−91·6 (5)
C(8)–C(9)–C(10)–C(11)	156·1 (4)	C(9)–C(10)–C(11)–C(12)	−177·0 (4)	C(10)–C(11)–C(12)–C(13)	170·3 (3)
C(11)–C(12)–C(13)–C(14)	−84·0 (4)	C(12)–C(13)–C(14)–C(1)	−29·5 (4)	C(13)–C(14)–C(1)–O(15)	16·2 (4)
C(14)–C(1)–O(15)–C(12)	4·4 (4)	C(1)–O(15)–C(12)–C(13)	−23·2 (4)	O(15)–C(12)–C(13)–C(14)	32·2 (4)
C(16)–C(1)–O(15)–C(12)	124·7 (4)	C(16)–C(1)–C(2)–C(3)	58·5 (5)	O(17)–C(2)–C(1)–O(15)	−179·7 (3)
O(17)–C(2)–C(1)–C(16)	−62·3 (5)	C(18)–C(5)–C(4)–C(3)	108·1 (5)	C(18)–C(5)–C(6)–C(7)	0·9 (8)
C(19)–C(9)–C(8)–C(7)	65·7 (6)	C(19)–C(9)–C(10)–C(11)	−0·4 (6)	C(22)–C(21)–C(12)–C(11)	−62·8 (5)
C(22)–C(21)–C(12)–O(15)	179·5 (4)	C(23)–C(21)–C(12)–C(11)	172·1 (4)	C(23)–C(21)–C(12)–O(15)	54·4 (5)

the molecular model. However, most of the phases calculated with the best packing were correct.

The structure was refined by isotropic and anisotropic block-diagonal least squares with programs of Domenicano, Spagna & Vaciago (1969). Scattering factors for C and O were from Cromer & Mann (1968) and for H from Hanson, Herman, Lea & Skillman (1964). The function minimized was $\sum w(|F_o| - |F_c|)^2$, with $w = (a + |F_o| + b|F_o|^2)^{-1}$. The H atoms were generated at the expected positions. Their B's (5 Å^2) and positional parameters were kept fixed. R and R_w were 0·057 and 0·076 respectively. The final atomic coordinates are reported in Table 1.*

Bond lengths and angles are given in Figs. 1 and 2. A schematic drawing of the molecular packing viewed along *c* is shown in Fig. 3.

Discussion. No unusual bond lengths and angles are observed. The epoxide ring is very nearly an equilateral triangle with the O atom protruding outside the macrocycle. C(1)–O(15)–C(12) is wider than 108°, as found in other cases, and the O atom points almost towards the centre of the molecule. The conformation of the molecule is described by the torsion angles given according to the convention of Klyne & Prelog (1960) in Table 2. C(14), C(1), O(15) and C(12) lie approximately in a plane, whereas C(13) is far from this plane, so that the tetrahydrofuran ring assumes an envelope conformation. The H atoms linked to C(3)–C(4) and to C(7)–C(8) are staggered. The widening of C(5)–C(6)–C(7) may be accounted for by the short distance between the C(18) methyl group and one of the H atoms of C(7). O(17) is *trans* and *gauche* with respect to O(15) and the C(16) methyl group, respectively. C(18), C(5) and C(6) define a plane almost perpendicular to the mean plane passing through the atoms of the macrocycle.

The crystal packing (Fig. 3) is mainly stabilized by hydrogen bonds, formed between O(17) and O(20) [2·829 (5) Å], which give rise to rows of molecules parallel to *b*. These rows are held together by strong van der Waals interactions which chiefly involve the isopropyl and methyl groups.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35686 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.