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3,3′-[Ethylenebis(aminomethylene)]di-2-bornanone

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Abstract. $C_{24}H_{36}N_2O_2$, $M_r = 384{\cdot}56$, tetragonal, $P4_32_12$, $a = 11{\cdot}143$ (3), $c = 19{\cdot}022$ (5) Å, $D_x = 1{\cdot}06$, $D_c = 1{\cdot}081$ Mg m^{-3}, $Z = 4$. Final $R = 0{\cdot}048$ for 999 reflections. The atoms of the ethylenediamine bridge are in a *gauche* conformation with the 2-bornanone moieties in an *anti* or *E* arrangement making intramolecular hydrogen bonding of the ketoenamine impossible. The bond lengths and angles are normal. The molecules are linked together by intermolecular N—H···O hydrogen bonds.

Introduction. The structures in solution of the condensation products between diamines and formylcamphor have been studied by Jensen & Larsen (1975, 1977) with different spectroscopic techniques (^{1}H, NMR, UV absorption and circular dichroism). From these measurements they obtained information about the relative distribution between the possible tautomers of the condensation products and, by application of the exciton theory to interpret the absorption and circular dichroism spectra, they deduced the relative orientations of the two formylcamphor chromophores in the Schiff bases. The structure analysis of the title compound was undertaken to see whether the structural conclusions obtained from the solution spectra could be confirmed by the structure in the solid state.

The space group and preliminary cell parameters were determined from photographs. Conditions for the reflections are: $h00$ observed for $h = 2n$; $00l$ observed for $l = 4n$; space group $P4_12_12$ or $P4_32_12$. An almost octahedral single crystal with maximum dimensions $0{\cdot}28 \times 0{\cdot}30 \times 0{\cdot}33$ mm was used for both cell refinement and data collection on a Picker FACS-1 diffractometer with graphite-monochromated Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å) at 295 K. By operation of the diffractometer in a θ–2θ scan mode at a rate of 1° min^{-1} in 2θ, intensities were recorded for one octant of reflections in the range $2{\cdot}4 \leq 2\theta \leq 127°$. The symmetry-related reflections were averaged to give 1183 independent reflections; the 999 reflections with $I/\sigma(I) \geq 2{\cdot}0$ were used for the analysis. Six standard reflections were measured after every 50 reflections and showed that no deterioration or misalignment had occurred. Lorentz and polarization corrections were applied. No correction was made for absorption [μ(Cu $K\alpha$) = $0{\cdot}50$ mm^{-1}]. The structure was solved by *MULTAN* (Germain, Main & Woolfson, 1971). A standard run assuming the space group $P4_12_12$ revealed all the non-hydrogen atoms. The absolute configuration of the 2-bornanone moiety has previously been determined by Allen & Rogers (1971). The structure was transformed to the enantiomeric space group $P4_32_12$ to obtain the correct absolute configuration.

Table 1. *Final positional parameters with e.s.d.'s in parentheses*

	x	y	z	U_{eq} (Å^2)*
C(1)	0·7935 (4)	0·1454 (4)	0·5328 (2)	0·067
C(2)	0·7536 (4)	0·0534 (4)	0·4775 (2)	0·062
C(3)	0·6906 (4)	−0·0374 (4)	0·5151 (2)	0·058
C(4)	0·6919 (4)	0·0008 (4)	0·5915 (2)	0·059
C(5)	0·8211 (5)	−0·0192 (5)	0·6175 (2)	0·072
C(6)	0·8920 (4)	0·0762 (5)	0·5761 (2)	0·075
C(7)	0·6850 (4)	0·1392 (4)	0·5840 (2)	0·067
C(8)	0·5671 (6)	0·1814 (5)	0·5522 (2)	0·085
C(9)	0·7019 (6)	0·2076 (5)	0·6533 (3)	0·086
C(10)	0·8322 (6)	0·2651 (5)	0·5036 (3)	0·097
C(11)	0·6481 (4)	−0·1395 (4)	0·4865 (2)	0·060
C(12)	0·5642 (4)	−0·3437 (4)	0·4898 (2)	0·070
N	0·5904 (3)	−0·2274 (3)	0·5197 (2)	0·065
O	0·7776 (3)	0·0603 (3)	0·41422 (12)	0·075
H(N)	0·577 (3)	−0·222 (3)	0·568 (2)	
H(4)	0·626 (3)	−0·035 (4)	0·620 (2)	
H(51)	0·848 (3)	−0·098 (3)	0·606 (2)	
H(52)	0·830 (3)	−0·012 (3)	0·673 (2)	
H(61)	0·951 (3)	0·039 (3)	0·544 (2)	
H(62)	0·940 (3)	0·136 (3)	0·606 (2)	
H(81)	0·575 (3)	0·267 (3)	0·534 (2)	
H(82)	0·502 (3)	0·154 (4)	0·581 (2)	
H(83)	0·544 (3)	0·141 (3)	0·512 (2)	
H(91)	0·770 (3)	0·194 (4)	0·674 (2)	
H(92)	0·708 (3)	0·301 (4)	0·641 (2)	
H(93)	0·638 (3)	0·184 (4)	0·684 (2)	
H(101)	0·850 (4)	0·317 (4)	0·543 (2)	
H(102)	0·903 (3)	0·251 (3)	0·473 (2)	
H(103)	0·764 (3)	0·298 (3)	0·472 (2)	
H(11)	0·659 (3)	−0·146 (3)	0·435 (2)	
H(121)	0·563 (3)	−0·335 (3)	0·432 (2)	
H(122)	0·488 (3)	−0·374 (3)	0·506 (2)	

* The equivalent isotropic thermal parameters were calculated from the expression $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

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The structure was refined by least squares, minimizing $\sum w(|F_o| - |F_c|)^2$. A difference synthesis calculated after anisotropic refinement showed the positions of the H atoms. They were given a fixed isotropic temperature factor $U = 0{\cdot}06$ Å^2 but their positional parameters were included in the refinement. The weights used in final cycles (Nielsen, 1977) were $w^{-1} = 3{\cdot}8 + 0{\cdot}04|F|$. Scattering factors were from Cromer & Mann (1968) for C, N and O, from Stewart, Davidson & Simpson (1965) for H. In the last cycle the maximum shift of parameters was 0·16σ; $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ and $R_w = [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2}$ were 0·048 and 0·046 respectively for the 999 observed reflections. The goodness of fit was $G = [\sum w(|F_o| - |F_c|)^2/(N - M)]^{1/2} = 2{\cdot}23$. The calculations were made with XRAY (1972). The final coordinates are shown in Table 1.*

Discussion. The atom numbering is illustrated in Fig. 1, which shows the molecule viewed parallel to the twofold axis. The arrangement of the N atoms of the ethylenediamine bridge is *gauche*, similar to the arrangement in the analogous condensation product between *R*-1,2-propanediamine and 2,4-pentanedione (Bernth, Larsen & Larsen, 1981). The conformation of the ketoenamine form of the formylcamphor groups can be described as an *anti* or *E* arrangement (Jensen & Larsen, 1977), making it impossible for the type of intramolecular hydrogen bond observed in the related 2,4-pentanedione condensation product to be formed. These stereochemical aspects of the structure are in agreement with the predictions of Jensen & Larsen (1975, 1977). Other structural features are shown in Table 2 which lists bond lengths, bond angles and

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

C(1)–C(2)	1·533 (6)	C(5)–H(51)	0·96 (4)
C(1)–C(10)	1·508 (7)	C(5)–H(52)	1·07 (3)
C(2)–C(3)	1·424 (6)	C(6)–H(61)	0·99 (4)
C(2)–O	1·236 (4)	C(6)–H(62)	1·02 (4)
C(3)–C(4)	1·514 (5)	C(8)–H(81)	1·02 (4)
C(3)–C(11)	1·347 (6)	C(8)–H(82)	0·96 (4)
C(4)–C(5)	1·539 (7)	C(8)–H(83)	0·93 (4)
C(4)–C(7)	1·550 (6)	C(9)–H(91)	0·88 (4)
C(5)–C(6)	1·540 (7)	C(9)–H(92)	1·07 (4)
C(6)–C(1)	1·576 (7)	C(9)–H(93)	0·96 (4)
C(7)–C(1)	1·555 (6)	C(10)–H(101)	0·97 (4)
C(7)–C(8)	1·521 (8)	C(10)–H(102)	0·99 (4)
C(7)–C(9)	1·534 (7)	C(10)–H(103)	1·04 (4)
C(11)–N	1·332 (6)	C(11)–H(11)	0·99 (3)
N–C(12)	1·445 (6)	N–H(N)	0·93 (4)
C(12)–C(12)′	1·502 (7)	C(12)–H(121)	1·10 (3)
C(4)–H(4)	0·99 (4)	C(12)–H(122)	0·97 (4)

C(6)–C(1)–C(2)	103·5 (3)	C(3)–C(4)–C(7)	101·0 (3)
C(6)–C(1)–C(10)	115·2 (4)	C(5)–C(4)–C(7)	102·7 (4)
C(2)–C(1)–C(10)	115·0 (4)	C(4)–C(5)–C(6)	102·5 (4)
C(6)–C(1)–C(7)	101·1 (3)	C(5)–C(6)–C(1)	104·3 (4)
C(2)–C(1)–C(7)	100·1 (3)	C(1)–C(7)–C(4)	93·6 (3)
C(7)–C(1)–C(10)	119·5 (4)	C(1)–C(7)–C(8)	114·1 (3)
C(1)–C(2)–C(3)	105·8 (3)	C(4)–C(7)–C(8)	112·8 (4)
C(1)–C(2)–O	124·3 (4)	C(1)–C(7)–C(9)	114·9 (4)
C(3)–C(2)–O	129·8 (4)	C(4)–C(7)–C(9)	114·2 (3)
C(2)–C(3)–C(4)	106·1 (3)	C(8)–C(7)–C(9)	107·1 (4)
C(2)–C(3)–C(11)	124·8 (3)	C(3)–C(11)–N	126·9 (4)
C(4)–C(3)–C(11)	128·9 (4)	C(11)–N–C(12)	124·8 (3)
C(3)–C(4)–C(5)	106·0 (3)	N–C(12)–C(12)′	111·9 (4)

N–C(12)–C(12)′–N′	−66·4 (4)
C(7)–C(1)–C(2)–C(3)	−34·8 (4)
C(2)–C(3)–C(4)–C(7)	34·1 (4)
C(3)–C(4)–C(7)–C(1)	−52·6 (4)
C(4)–C(7)–C(1)–C(2)	52·5 (3)
C(6)–C(1)–C(7)–C(4)	−53·6 (4)
C(2)–C(3)–C(4)–C(5)	−72·7 (4)
C(3)–C(4)–C(5)–C(6)	68·0 (4)
C(3)–C(11)–N–C(12)	−169·0 (4)
C(1)–C(2)–C(3)–C(4)	0·6 (5)
C(4)–C(5)–C(6)–C(1)	2·5 (4)
C(7)–C(4)–C(5)–C(6)	−37·5 (4)
C(7)–C(1)–C(6)–C(5)	33·0 (4)
C(1)–C(7)–C(4)–C(5)	56·7 (3)
C(6)–C(1)–C(2)–C(3)	69·3 (4)
C(5)–C(6)–C(1)–C(2)	−70·3 (4)

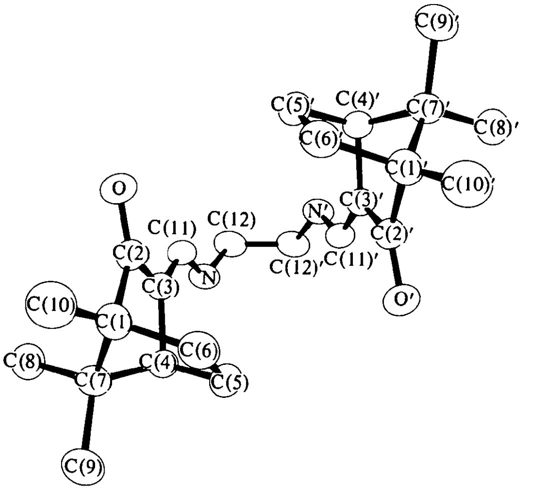

Fig. 1. A view of the title compound parallel to the molecular twofold axis.

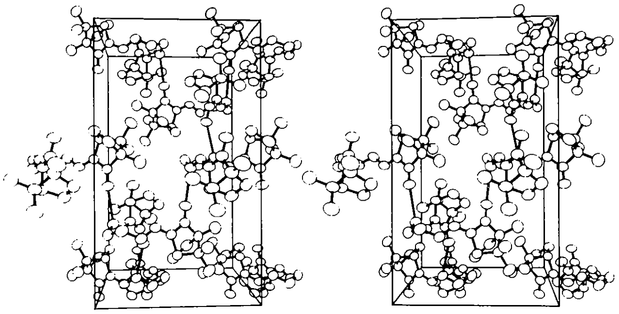

Fig. 2. Stereoscopic drawing of the packing viewed along *a*. The open bonds designate hydrogen bonds.

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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 35669 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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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