C(15)=C(20)–C(21) is in a nearly *cis* arrangement, the torsional angle C(20)–C(15)–C(16)–O(16) of 44·0 (6)° agreeing well with the values of 40·5 (5) and 46·2 (3)° found for hygrophylline and senecionine respectively.

The hydroxyl group at C(2) and the N atom of a neighbouring molecule are linked by an intermolecular hydrogen bond. The O(2)···N(4), O(2)–H(3) and N(4)···H(3) distances are 2·735 (6), 0·86 and 1·88 Å and the angle O(2)–H(3)···N(4) is 171°. Similar intermolecular interactions involving a hydroxyl substituent at C(12), instead of at C(2) as in rosmarinine, are observed in senecionine, 2·827 (4), 0·77 (5), 2·07 (5) Å and 167 (3)°, and retrorsine (Coleman, Coucourakis & Pretorius, 1980), 2·85 (1), 0·98 (7), 1·90 (7) Å and 162°.

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Structure of Econazole

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Abstract. 1-{2-[(4-Chlorophenyl)methoxy]-2-(2,4-dichlorophenyl)ethyl}-1*H*-imidazole, $C_{18}H_{15}Cl_3N_2O$, $M_r = 381{\cdot}7$, monoclinic, $P2_1/c$, $a = 8{\cdot}410\ (1)$, $b = 11{\cdot}084\ (1)$, $c = 19{\cdot}657\ (2)$ Å, $\beta = 96{\cdot}24\ (1)°$, $V = 1821{\cdot}3$ Å^3, $Z = 4$, $D_m = 1{\cdot}42$, $D_x = 1{\cdot}39$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 47{\cdot}08$ cm^{-1}, $F(000) = 784$, $T = 291$ K, final $R = 0{\cdot}040$ for 2083 unique observed reflections. Each of the three ring systems is essentially planar, with the *p*-chlorophenyl ring twisted at an angle of approximately 60° to the other two ring systems which are almost coplanar. Intra- and intermolecular distances and angles are within the values recorded for similar compounds.

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Introduction. Econazole is a prime member of the group of imidazole derivatives (Godefroi, Heeres, Van Cutsem & Janssen, 1969) which have achieved prominence as antimycotic agents (Drouhet, 1978). Like its close analogue miconazole, which differs in having an additional Cl at C(19) in the benzyloxy moiety, econazole has a wide antifungal spectrum (Thienpont, Van Cutsem, Van Nueten, Niemegeers & Marsboom, 1975) and is of particular clinical use in the topical treatment of superficial mycoses. As the racemic free-base forms of both econazole and miconazole are poorly soluble in aqueous systems, they were selected as models for a study of crystal growth in pharmaceutical semi-solid formulations (Pearson, Shankland, Salole & Johnson, 1985). Since the crystal-

lographic structure of miconazole had been elucidated (Peeters, Blaton & De Ranter, 1979) it was considered of interest to determine the structure of econazole.

Experimental. Colourless, thin needle-shaped crystals grown by slow evaporation under nitrogen from aqueous ethanol, crystal *ca* 1·2 × 0·6 × 0·4 mm used in data collection, CAD-4 diffractometer. Preliminary Weissenberg photographs indicated crystals to be monoclinic, $P2_1/c$. D_m by flotation. 3447 independent intensities, θ limit 70°, $\omega/2\theta$ scan. Two standard intensities used to monitor variations in intensity data: <3% variation observed. Least-squares technique based on 25 reflections, $\theta > 15°$, used to refine lattice parameters. No absorption correction. h 0 to 10, k 0 to 13, l −23 to 23. Structure solution by direct methods with *MITHRIL* (Gilmore, 1984). Full-matrix least-squares refinement on F of coordinates and anisotropic thermal parameters for non-H atoms converged to R and wR of 0·040 and 0·040 with unit weights. H-atom coordinates determined from difference Fourier synthesis and subsequently refined isotropically in final two cycles of least squares. 2083 reflections, $I \geq 3{\cdot}0\sigma_I$, used. $\Delta_{max}/\sigma = 0{\cdot}19$; max. and min. heights in final difference Fourier synthesis = 0·21 and −0·27 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations on a Gould SEL 32/27 computer using Glasgow *GX* package (Mallinson & Muir, 1985).

Discussion. Final positional and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths, bond angles and selected torsion angles are given in Table 2. An *ORTEP* (Johnson, 1976) diagram, Fig. 1, illustrates the numbering scheme for the molecule.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43077 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

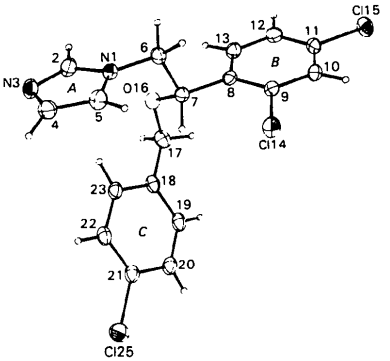

Fig. 1. A perspective view of the molecule showing the numbering scheme and vibrational ellipsoids (45% probability level).

Table 1. *Final positional parameters and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Cl(14)	0·68667 (13)	0·20372 (11)	0·99832 (5)	0·076
Cl(15)	0·32708 (13)	0·52974 (11)	1·11047 (7)	0·085
Cl(25)	0·88476 (15)	−0·40617 (11)	1·06972 (7)	0·090
O(16)	0·9366 (3)	0·1549 (3)	1·2026 (1)	0·063
N(1)	1·1880 (3)	0·1962 (3)	1·1198 (2)	0·059
N(3)	1·3989 (4)	0·0907 (3)	1·1618 (2)	0·073
C(2)	1·2913 (5)	0·1691 (4)	1·1754 (2)	0·070
C(4)	1·3643 (5)	0·0662 (4)	1·0941 (3)	0·073
C(5)	1·2343 (5)	0·1289 (4)	1·0668 (2)	0·069
C(6)	1·0455 (5)	0·2727 (4)	1·1193 (3)	0·066
C(7)	0·8997 (4)	0·1996 (4)	1·1354 (2)	0·053
C(8)	0·7534 (4)	0·2804 (3)	1·1292 (2)	0·051
C(9)	0·6525 (4)	0·2910 (3)	1·0688 (2)	0·051
C(10)	0·5212 (4)	0·3664 (4)	1·0625 (2)	0·057
C(11)	0·4924 (4)	0·4342 (4)	1·1183 (2)	0·058
C(12)	0·5888 (5)	0·4284 (4)	1·1793 (2)	0·064
C(13)	0·7191 (5)	0·3514 (4)	1·1838 (2)	0·062
C(17)	0·8326 (6)	0·0616 (4)	1·2201 (3)	0·072
C(18)	0·8453 (4)	−0·0534 (4)	1·1813 (2)	0·057
C(19)	0·7142 (5)	−0·1260 (4)	1·1646 (3)	0·069
C(20)	0·7243 (5)	−0·2338 (5)	1·1318 (3)	0·070
C(21)	0·8706 (5)	−0·2711 (4)	1·1127 (2)	0·061
C(22)	1·0022 (5)	−0·2001 (4)	1·1286 (2)	0·064
C(23)	0·9910 (5)	−0·0922 (4)	1·1617 (2)	0·061

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

Cl(14)–C(9)	1·738 (4)	Cl(15)–C(11)	1·741 (4)
Cl(25)–C(21)	1·730 (5)	O(16)–C(7)	1·414 (5)
O(16)–C(17)	1·420 (6)	N(1)–C(2)	1·354 (6)
N(1)–C(5)	1·372 (6)	N(1)–C(6)	1·467 (5)
N(3)–C(2)	1·303 (6)	N(3)–C(4)	1·359 (6)
C(4)–C(5)	1·356 (7)	C(6)–C(7)	1·530 (6)
C(7)–C(8)	1·516 (6)	C(8)–C(9)	1·388 (6)
C(8)–C(13)	1·386 (6)	C(9)–C(10)	1·380 (6)
C(10)–C(11)	1·373 (6)	C(11)–C(12)	1·375 (7)
C(12)–C(13)	1·384 (6)	C(17)–C(18)	1·495 (7)
C(18)–C(19)	1·376 (6)	C(18)–C(23)	1·392 (6)
C(19)–C(20)	1·365 (8)	C(20)–C(21)	1·388 (6)
C(21)–C(22)	1·366 (6)	C(22)–C(23)	1·370 (7)
C(7)–O(16)–C(17)	113·8 (4)	C(2)–N(1)–C(5)	106·4 (4)
C(2)–N(1)–C(6)	125·8 (4)	C(5)–N(1)–C(6)	127·5 (4)
C(2)–N(3)–C(4)	104·8 (4)	N(1)–C(2)–N(3)	112·4 (4)
N(3)–C(4)–C(5)	111·3 (4)	N(1)–C(5)–C(4)	105·2 (4)
N(1)–C(6)–C(7)	111·4 (4)	O(16)–C(7)–C(6)	106·2 (4)
O(16)–C(7)–C(8)	112·0 (4)	C(6)–C(7)–C(8)	109·4 (4)
C(7)–C(8)–C(9)	122·2 (4)	C(7)–C(8)–C(13)	120·6 (4)
C(9)–C(8)–C(13)	117·1 (4)	Cl(14)–C(9)–C(8)	119·8 (3)
Cl(14)–C(9)–C(10)	117·7 (3)	C(8)–C(9)–C(10)	122·5 (4)
C(9)–C(10)–C(11)	117·9 (4)	Cl(15)–C(11)–C(10)	118·1 (4)
Cl(15)–C(11)–C(12)	119·7 (4)	C(10)–C(11)–C(12)	122·2 (4)
C(11)–C(12)–C(13)	118·2 (4)	C(8)–C(13)–C(12)	122·1 (4)
O(16)–C(17)–C(18)	114·7 (4)	C(17)–C(18)–C(19)	121·4 (4)
C(17)–C(18)–C(23)	121·3 (4)	C(19)–C(18)–C(23)	117·3 (4)
C(18)–C(19)–C(20)	122·4 (5)	C(19)–C(20)–C(21)	119·4 (5)
Cl(25)–C(21)–C(20)	120·0 (4)	Cl(25)–C(21)–C(22)	120·9 (4)
C(20)–C(21)–C(22)	119·2 (5)	C(21)–C(22)–C(23)	121·0 (4)
C(18)–C(23)–C(22)	120·7 (4)		
C(17)–O(16)–C(7)–C(6)	−165·5 (3)	C(17)–O(16)–C(7)–C(8)	75·2 (4)
C(7)–O(16)–C(17)–C(18)	66·9 (4)		
C(2)–N(1)–C(6)–C(7)	−87·0 (5)		
C(5)–N(1)–C(6)–C(7)	85·9 (5)		
N(1)–C(6)–C(7)–O(16)	63·0 (4)	N(1)–C(6)–C(7)–C(8)	−175·9 (5)
C(6)–C(7)–C(8)–C(9)	92·2 (5)	C(6)–C(7)–C(8)–C(13)	−85·0 (5)
O(16)–C(17)–C(18)–C(23)	34·2 (4)	O(16)–C(17)–C(18)–C(19)	−146·8 (6)

Each of the three ring systems in the molecule is almost planar with a maximum deviation of 0·006 (4) Å from the least-squares plane. The imidazole ring, *A*, and the 2,4-dichlorophenyl ring, *B*, lie close to the same plane [dihedral angle 4·4 (2)°] whilst the *p*-chlorophenyl ring, *C*, is at an angle of 60·7 (4)° to that plane. This is a similar situation to that observed for miconazole except that ring *C* is found to be nearly perpendicular to the almost coplanar *A*,*B* ring system.

The absence of a Cl atom at C(19), compared to miconazole, allows several changes in conformation whereby ring *B* rotates through 180° such that Cl(14) is on the same side of the molecule as the *p*-chlorophenyl ring with the Cl atom adjacent to the ring nucleus. This situation confers a certain restriction on the degree of rotation of ring *C* about the O(16)–C(17) bond which is observed in the torsion angles C(7)–O(16)–C(17)–C(18), 67·0 (4)°, and O(16)–C(17)–C(18)–C(23), 34·2 (4)°, when compared to the values of −179·8 (8) and 0·9 (16)° found in miconazole. Whether these subtle conformational differences affect the pharmacological activity of the compound is difficult to assess, mainly because data obtained from *in vitro* experiments are derived from a number of differing techniques and test organisms. Recent experiments (Brasseur, Vandenbosch, Marichal, Van den Bossche & Ruysschaert, 1982) show that when imidazole derivatives are inserted in model lipid bilayers the two phenyl residues are maintained in the hydrophobic phase with the imidazole residue orientated towards the hydrophilic phase, which may partly be the mechanism by which they affect membrane fluidity.

Intra- and intermolecular distances and angles are within the values recorded for similar compounds.

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Structure of 5β,24-Cyclofriedelan-3-one, a Novel Hexacyclic Triterpenoid in Space Group *P*1

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Abstract. $C_{30}H_{48}O$, $M_r = 424·7$, triclinic, $P1$, $a = 6·259$ (3), $b = 7·351$ (1), $c = 14·382$ (3) Å, $\alpha = 85·31$ (1), $\beta = 87·25$ (2), $\gamma = 65·89$ (2)°, $V = 601·9$ Å^3, $Z = 1$, $D_x = 1·17$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 0·63$ cm^{-1}, $F(000) = 236$, $T = 291$ K, final $R = 0·057$ for 1607 unique reflections. The molecule adopts an extended *S*, stretched, conformation for the chain of five six-membered rings. The incorporation of a methyl group of a typical pentacyclic triterpenoid into a cyclopropane ring is uncommon and provides the first example of a cyclofriedelane. The elucidation of the crystal structure required the use of three distinct program packages.

Introduction. The triterpenoid ketone (1), m.p. 598 K, $[\alpha]_D$ +54·9°, was isolated from the leaves and stems of *Euphorbia nerifolia* (Anjaneyulu & Row, 1965). It was originally assigned (Anjaneyulu, Row, Subrahmanyam

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