than that for (2). The results of this study clearly show that the symmetry and O···O distances of the short hydrogen bonds depend on the crystallographic environment.

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Structure of 28-Hydroxyfriedelan-3-one

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Abstract. $C_{30}H_{50}O_2$, $M_r = 442{\cdot}7$, orthorhombic, $P2_12_12_1$, $a = 14{\cdot}017$ (5), $b = 28{\cdot}671$ (11), $c = 6{\cdot}428$ (2) Å, $V = 2583{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}132$ cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 4{\cdot}866$ cm^{-1}, $F(000) = 984$, $T = 296$ K, $R = 0{\cdot}075$ for 2430 observed reflections. The compound was isolated from the plant *Pristimera grahamii.* The molecule consists of five fused six-membered rings *A, B, C, D* and *E.* The ring junctions *A/B, B/C, C/D* and *D/E* are, respectively, *cis, trans, trans, gauche.* Rings *A, B, C* are in a chair conformation. Rings *D* and *E* are, respectively, in twist-boat and distorted chair conformations. The packing of the molecules is due to van der Waals forces and there are no intermolecular hydrogen bonds.

Introduction. The title compound was isolated for the first time from the plant *Pristimera grahamii.* The plant is said to be used as an anti-cancer drug and also for protection in repelling insect and microbial attack, as an indigenous medicine in India. This structure investigation has been undertaken to interpret the spectral data.

Experimental. Transparent, colourless crystals were grown from benzene solution. Crystal dimensions 0·5 × 0·4 × 0·45 mm, CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions were determined by least-squares refinement using 2θ values of 25 medium-angle reflections ($25° < \theta < 35°$). Intensity data were collected at room temperature by $\omega/2\theta$ scan technique up to $2\theta \simeq 170°$ with h 0→16, k 0→34 and l 0→7. Two standard reflections monitored every 100 measurements, variation less than 2%; 2648 unique reflections measured of which 2430 intensities observed $[I > 3\sigma(I)]$. The intensities were corrected for Lp effects; no absorption corrections. Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Hydrogen atoms located on a difference density map. Positional parameters of all atoms and anisotropic thermal parameters of non-hydrogen atoms refined by full-matrix least-squares

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors for non-hydrogen atoms, with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}(Å^2)
C(1)	0·7507 (4)	0·0979 (2)	0·2615 (9)	4·1 (1)
C(2)	0·7740 (4)	0·0567 (2)	0·1222 (10)	4·4 (1)
C(3)	0·6889 (4)	0·0276 (2)	0·0824 (9)	4·1 (1)
C(4)	0·6020 (4)	0·0531 (2)	0·0064 (9)	3·8 (1)
C(5)	0·5737 (3)	0·0903 (2)	0·1752 (8)	3·5 (1)
C(6)	0·4862 (3)	0·1178 (2)	0·0974 (10)	3·8 (1)
C(7)	0·4680 (3)	0·1630 (2)	0·2202 (10)	4·1 (1)
C(8)	0·5560 (3)	0·1944 (1)	0·2062 (8)	3·0 (1)
C(9)	0·6426 (3)	0·1707 (2)	0·3167 (7)	3·1 (1)
C(10)	0·6612 (3)	0·1242 (1)	0·1926 (7)	3·0 (1)
C(11)	0·7303 (3)	0·2024 (2)	0·2925 (9)	3·7 (1)
C(12)	0·7118 (3)	0·2536 (2)	0·3515 (8)	3·5 (1)
C(13)	0·6301 (3)	0·2762 (2)	0·2296 (7)	3·0 (1)
C(14)	0·5375 (3)	0·2467 (2)	0·2645 (7)	3·0 (1)
C(15)	0·4575 (4)	0·2654 (2)	0·1149 (10)	4·1 (1)
C(16)	0·4651 (4)	0·3177 (2)	0·0592 (9)	4·2 (1)
C(17)	0·5225 (3)	0·3510 (2)	0·2004 (8)	3·5 (1)
C(18)	0·6120 (3)	0·3271 (2)	0·3013 (7)	3·1 (1)
C(19)	0·7045 (4)	0·3577 (2)	0·2719 (10)	4·5 (1)
C(20)	0·6937 (5)	0·4096 (2)	0·3169 (10)	5·0 (1)
C(21)	0·6060 (5)	0·4295 (2)	0·1955 (1)	5·7 (1)
C(22)	0·5535 (4)	0·3925 (2)	0·0712 (9)	4·7 (1)
C(23)	0·5203 (4)	0·0212 (2)	−0·0626 (10)	4·9 (1)
C(24)	0·5514 (5)	0·0642 (2)	0·3799 (10)	5·1 (1)
C(25)	0·6311 (4)	0·1617 (2)	0·5543 (8)	4·4 (1)
C(26)	0·5014 (4)	0·2500 (2)	0·4903 (9)	3·9 (1)
C(27)	0·6615 (4)	0·2752 (2)	−0·0051 (9)	4·1 (1)
C(28)	0·4547 (4)	0·3685 (2)	0·3749 (9)	4·3 (1)
O(29)	0·3767 (3)	0·3934 (2)	0·2924 (8)	6·4 (1)
C(30)	0·7863 (6)	0·4325 (2)	0·2454 (10)	7·9 (1)
C(31)	0·6843 (6)	0·4180 (2)	0·5492 (10)	6·3 (1)
O(32)	0·6897 (4)	−0·0145 (1)	0·1165 (7)	5·6 (1)

method. Final $R = 0{\cdot}075$ (high value due to poor crystal quality) and $wR = 0{\cdot}081$. $S = 2{\cdot}5$ for 2430 observed reflections. $w = K/[\sigma^2|F_o| + m|F_o|^2]$ ($K = 6{\cdot}171$, $m = 0{\cdot}00038$). Ratio of final shift to e.s.d. $< 0{\cdot}1$; final $\Delta\rho$ excursions $< 0{\cdot}57$ e Å^{-3}. The atomic scattering factors used for all the atoms were as provided in the *SHELX*76 program. Computer programs: *SHELX*76 used for refinement (Sheldrick, 1976) and molecular illustrations drawn using *MOLDRAW* (Ugliengo, Borzani & Viterbo, 1988); *PARST* (Nardelli, 1983) for geometrical calculations.

Discussion. Table 1* lists the final atomic coordinates and equivalent isotropic temperature factors of non-hydrogen atoms. The numbering scheme and the endocyclic torsion angles are given in Fig. 1. The bond lengths and angles are given in Table 2. The C—C bond lengths range from 1·478 (8) to 1·577 (7) Å with a mean value of 1·543 Å. The significant deviation from this mean may be due to the substitution at different C atoms (Gzella, Zaprutro & Wrzeciono, 1987). The endocyclic bond angles for tetrahedral atoms in rings *A*, *B*, *C*, *D* and *E* vary from 105·5 (3) to 121·1 (5)° with a mean value of 111·0°. This value compares well with that of a related compound (Hoge & Nordman, 1974). From Fig. 1 it is clear that *A*, *B*, and *C* take a chair conformation. The *D* ring is in a twist-boat conformation (Hendrickson, 1964). Ring *E* is a distorted chair. The molecule is bent progressively from ring *E* to ring *A* as shown by the angles between the mean planes

Table 2. *Bond lengths* (Å) *and angles* (°) *involving non-hydrogen atoms with e.s.d.'s in parentheses*

C(1)–C(2)	1·518 (8)	C(12)–C(13)	1·531 (7)
C(1)–C(10)	1·529 (7)	C(13)–C(14)	1·565 (7)
C(2)–C(3)	1·478 (8)	C(13)–C(18)	1·551 (8)
C(3)–C(4)	1·502 (8)	C(13)–C(27)	1·572 (7)
C(3)–O(32)	1·227 (6)	C(14)–C(15)	1·571 (7)
C(4)–C(5)	1·572 (8)	C(14)–C(26)	1·540 (7)
C(4)–C(23)	1·531 (8)	C(15)–C(16)	1·545 (8)
C(5)–C(6)	1·541 (7)	C(16)–C(17)	1·543 (8)
C(5)–C(10)	1·569 (6)	C(17)–C(18)	1·570 (6)
C(5)–C(24)	1·546 (8)	C(17)–C(22)	1·515 (8)
C(6)–C(7)	1·539 (8)	C(17)–C(28)	1·553 (7)
C(7)–C(8)	1·530 (6)	C(18)–C(19)	1·577 (7)
C(8)–C(9)	1·562 (6)	C(19)–C(20)	1·523 (8)
C(8)–C(14)	1·567 (6)	C(20)–C(21)	1·564 (10)
C(9)–C(10)	1·575 (6)	C(20)–C(30)	1·525 (16)
C(9)–C(11)	1·537 (7)	C(20)–C(31)	1·518 (14)
C(9)–C(25)	1·557 (7)	C(21)–C(22)	1·518 (9)
C(11)–C(12)	1·538 (8)	C(28)–O(29)	1·409 (7)

C(2)–C(1)–C(10)	112·9 (5)	C(12)–C(13)–C(14)	108·5 (4)
C(1)–C(2)–C(3)	111·6 (5)	C(18)–C(13)–C(27)	110·4 (4)
C(2)–C(3)–O(32)	121·1 (5)	C(14)–C(13)–C(27)	111·1 (4)
C(2)–C(3)–C(4)	115·8 (5)	C(14)–C(13)–C(18)	109·3 (4)
C(4)–C(3)–O(32)	123·0 (5)	C(8)–C(14)–C(13)	110·2 (4)
C(3)–C(4)–C(23)	114·2 (5)	C(13)–C(14)–C(26)	112·0 (4)
C(3)–C(4)–C(5)	108·1 (4)	C(13)–C(14)–C(15)	108·6 (4)
C(5)–C(4)–C(23)	114·6 (4)	C(8)–C(14)–C(26)	109·8 (4)
C(4)–C(5)–C(24)	108·1 (4)	C(8)–C(14)–C(15)	107·3 (4)
C(4)–C(5)–C(10)	105·8 (4)	C(15)–C(14)–C(26)	108·7 (4)
C(4)–C(5)–C(6)	108·9 (4)	C(14)–C(15)–C(16)	115·1 (5)
C(10)–C(5)–C(24)	113·4 (4)	C(15)–C(16)–C(17)	120·0 (5)
C(6)–C(5)–C(24)	111·3 (4)	C(16)–C(17)–C(28)	107·8 (4)
C(6)–C(5)–C(10)	109·2 (4)	C(16)–C(17)–C(22)	108·2 (4)
C(5)–C(6)–C(7)	113·3 (4)	C(16)–C(17)–C(18)	112·9 (4)
C(6)–C(7)–C(8)	109·4 (4)	C(22)–C(17)–C(28)	108·5 (4)
C(7)–C(8)–C(14)	114·6 (4)	C(18)–C(17)–C(28)	109·4 (4)
C(7)–C(8)–C(9)	110·1 (3)	C(18)–C(17)–C(22)	109·9 (4)
C(9)–C(8)–C(14)	115·8 (4)	C(13)–C(18)–C(17)	114·7 (4)
C(8)–C(9)–C(25)	115·9 (4)	C(17)–C(18)–C(19)	111·4 (4)
C(8)–C(9)–C(11)	108·6 (4)	C(13)–C(18)–C(19)	110·7 (4)
C(8)–C(9)–C(10)	105·5 (3)	C(18)–C(19)–C(20)	116·0 (5)
C(11)–C(9)–C(25)	106·3 (4)	C(19)–C(20)–C(31)	110·5 (5)
C(10)–C(9)–C(25)	111·9 (4)	C(19)–C(20)–C(30)	106·2 (5)
C(10)–C(9)–C(11)	108·5 (4)	C(19)–C(20)–C(21)	109·8 (5)
C(5)–C(10)–C(9)	115·5 (3)	C(30)–C(20)–C(31)	107·6 (6)
C(1)–C(10)–C(9)	114·0 (4)	C(21)–C(20)–C(31)	111·4 (6)
C(1)–C(10)–C(5)	110·9 (4)	C(21)–C(20)–C(30)	111·2 (5)
C(9)–C(11)–C(12)	113·9 (4)	C(20)–C(21)–C(22)	112·9 (5)
C(11)–C(12)–C(13)	113·8 (4)	C(17)–C(22)–C(21)	113·5 (5)
C(12)–C(13)–C(27)	105·9 (4)	C(17)–C(28)–O(29)	111·5 (4)
C(12)–C(13)–C(18)	111·6 (4)		

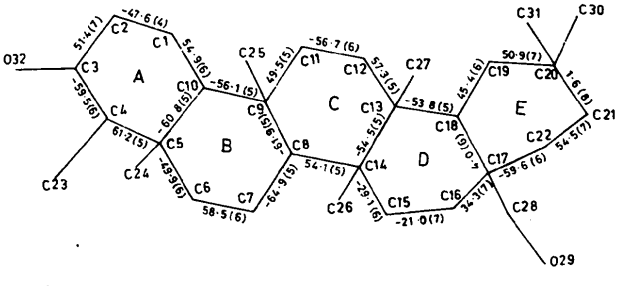

Fig. 1. Numbering of atoms and endocyclic torsion angles (°).

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and angles involving H atoms and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51643 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

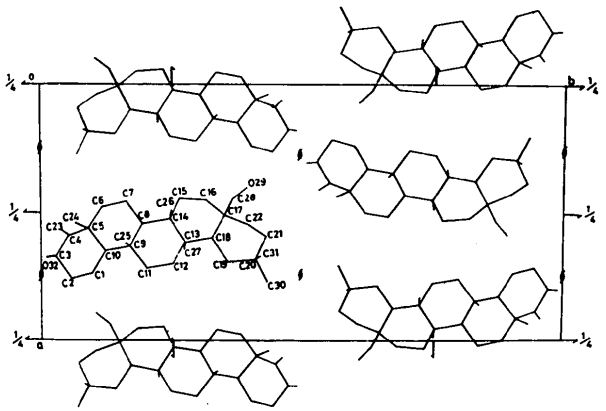

Fig. 2. Projection of the crystal structure along the *c* axis.

of these rings (*A–B* 9·4, *B–C* 16·4, *C–D* 13·4, *D–E* 13·5°). The ring junctions *A/B*, *B/C*, *C/D* and *D/E* are, respectively, *cis*, *trans*, *trans*, *gauche*. The *D* and *E* rings are considerably distorted to relieve the repulsive interaction between the C(25) and C(24) substituents which occupy the axial positions of the *D* and *E* rings (Nakai & Shiro, 1987). The non-bonded distances between C atoms of diaxial methyl groups C(24)···C(25) and C(25)···C(26) are 3·212 (8) and 3·141 (8) Å, and are larger than the values expected for non-distorted rings. A projection of the crystal structure is shown in Fig. 2. The shortest intermolecular distance [O(29)–H(29)···O(32) $(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$ = 2·860 (6) Å] implies possible hydrogen bonding but the angle around H is only 88 (4)°. Other than this, the crystal structure is stabilized by van der Waals forces.

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Ethyl (*Z*)-3-(2-Dimethylamino-4-methyl-6-oxo-6*H*-1,3-oxazin-5-yl)but-2-enoate (1) and Ethyl (*E*)-3-(2-Dimethylamino-6-oxo-4-phenyl-6*H*-1,3-oxazin-5-yl)but-2-enoate (2)

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Abstract. (1): $C_{13}H_{18}N_2O_4$, $M_r = 266{\cdot}3$, monoclinic, $P2_1/n$, $a = 6{\cdot}595$ (1), $b = 11{\cdot}708$ (1), $c = 18{\cdot}307$ (2) Å, $\beta = 97{\cdot}17$ (1)°, $V = 1402{\cdot}5$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}261$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}88$ cm^{-1}, $F(000) = 568$, room temperature, final $R = 0{\cdot}047$ for 1844 observed reflections. (2): $C_{18}H_{20}N_2O_4$, $M_r = 328{\cdot}4$, monoclinic, $P2_1/n$, $a = 10{\cdot}535$ (1), $b = 9{\cdot}454$ (1), $c = 17{\cdot}201$ (2) Å, $\beta = 91{\cdot}27$ (1)°, $V = 1712{\cdot}8$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}273$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}85$ cm^{-1}, $F(000) = 696$, room temperature, final $R = 0{\cdot}046$ for 2225 observed reflections. The 2-amino-6*H*-1,3-oxazin-6-one nuclei of the two molecules are very similar; this probably means that the different reactivity of the *Z* and *E* isomers is only due to steric reasons. Distances and angles along the butenoic chain are quite different in (1) and (2) because of the *Z* and *E* configuration.

Introduction. Becalli & Marchesini (1987) have studied the synthesis and reactivity of 2-dimethylamino-6*H*-1,3-oxazin-6-ones. Pilati (1988) (hereinafter P88) has shown that the reactivity of these compounds mainly depends on electronic reasons, as evidenced by significant variations in the 1,3-oxazin-6-one geometry. Becalli & Marchesini (1988) have recently extended their work on 5-vinyl substituted molecules; in particular they found that the *Z* and *E* isomers of $-CR^1{=}CH{-}CO_2Et$ derivatives have very different reactivity. For example, *E* compounds react very quickly by pyrolysis, according to the following

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