

for the H atoms are from Stewart, Davidson & Simpson (1965). The values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974). Positional and thermal parameters for non-H atoms are listed in Table 1,\* while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labelling scheme is shown in Fig. 1. The least-squares-planes program was supplied by Cordes (1983); other computer programs are listed in Gadol & Davis (1982).

**Related literature.** (1) is a bicyclic intermediate in a synthetic route aimed at preparing the tricyclic carbon framework of the taxane diterpenes (Martin, White & Wagner, 1982). The structures of related [5.3.1]undecenes have been reported (Lynch, Fishpaugh, Martin & Davis, 1990; Lynch, Tanaka, Fishpaugh, Martin & Davis, 1990). The alkene moiety in these structures is also highly distorted.

\* Lists of structure factors, anisotropic thermal parameters, torsion angles, least-squares planes, unit-cell packing diagram and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53032 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of the Eudesmanolide Gazaniolide\*

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**Abstract.** C<sub>15</sub>H<sub>18</sub>O<sub>2</sub>,  $M_r = 230.31$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.129$  (1),  $b = 11.126$  (2),  $c = 16.112$  (2) Å,  $V = 1278.0$  (6) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.197$  g cm<sup>-3</sup>, at 293 (1) K,  $\lambda(\text{Cu } K\alpha) = 1.54184$  Å,  $\mu = 5.8$  cm<sup>-1</sup>,  $F(000) = 496$ , 1535 unique reflections measured, final  $R = 0.038$  over 1217 reflections having  $I > 1.0\sigma(I)$ . This sesquiterpene lactone of the eudesmanolide class, extracted from the roots of the plant *Rudbeckia subtomentosa*, has an unusual 1,3-cyclohexadiene ring which is highly susceptible to air

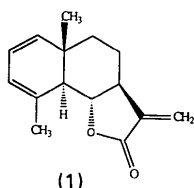
oxidation. The two six-membered rings are *trans*-fused, with the saturated ring in the chair conformation and the ring containing the diene in the half-chair conformation. The pseudo twofold axis of the half chair bisects the diene, and the torsion angle about the central bond of the diene is  $-16.1$  (4)°.

**Experimental.** A colorless, nearly isometric crystal of gazaniolide, (1),  $0.35 \times 0.38 \times 0.40$  mm, was mounted in a glass capillary in random orientation on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite crystal incident beam monochromator. Cell constants were obtained from least-squares refinement of the setting angles of 25 reflections in the range  $21 < \theta < 27^\circ$ . From systematic absences

\* *Chemical Abstracts* name: [3aS-(3a $\alpha$ ,5a $\beta$ ,9a $\alpha$ ,9b $\beta$ )]-3a-,4,5a,9a,9b-hexahydro-5a,9-dimethyl-3-methylenenaphtho[1,2-*b*]furan-2(3H)-one.

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( $h00$ ,  $h$  odd,  $0k0$ ,  $k$  odd,  $00l$ , odd) the space group was determined to be  $P2_12_12_1$ .



Two octants of data were collected using the  $\theta$ - $2\theta$  scan technique, variable scan rates from  $0.72$  to  $3.30^\circ \text{ min}^{-1}$  ( $h = 0$  to  $8$ ,  $k = 0$  to  $13$ ,  $l = -20$  to  $20$ ) to a maximum  $2\theta$  of  $150^\circ$ . A total of  $2978$  reflections was measured, of which  $1535$  were unique and not systematically absent. Redundant reflections were averaged with  $R_{\text{int}} = 0.025$ . Three representative reflections ( $200$ ,  $020$ ,  $006$ ) were measured every  $10\,000$  s, and only random variations were observed. Lorentz and polarization corrections were applied. An empirical absorption correction based on a series of  $\psi$  scans was applied to the data; relative transmission coefficients ranged from  $0.949$  to  $0.998$  with an average value of  $0.975$ .

The structure was solved by direct methods (Mair., Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Hydrogen atoms were located from difference maps and refined isotropically. The structure was refined by full-matrix least squares where the function minimized was  $\sum w(|F_o| - |F_c|)^2$  and the weight is  $w = 4F_o^2/\sigma^2(F_o^2)$ . The variance of the structure amplitude is defined as  $\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/Lp^2$  where  $S$  is the scan rate,  $C$  is the total integrated peak count,  $R$  is the ratio of scan time to background counting time,  $B$  is the total background count,  $Lp$  is the Lorentz-polarization factor, and  $p$  was set to  $0.020$ .

Scattering factors, including anomalous-dispersion terms, were taken from the *International Tables for X-ray Crystallography* (1974, Vol. IV). Only the  $1217$  reflections having  $I > 1\sigma(I)$  were used in the refinement. The final cycle of refinement included  $227$  variable parameters and converged (largest  $\Delta/\sigma = 0.03$ ) with  $R = 0.038$ ,  $wR = 0.038$ ,  $R(\text{all}) = 0.057$ , and  $S = 1.285$ . A secondary-extinction coefficient refined to a value of  $g = 3.31(12) \times 10^{-6}$ , where the correction factor  $(1 + gI_c)^{-1}$  was applied to  $F_c$ . There were no correlation coefficients greater than  $0.50$ . The maximum value in the final difference Fourier was  $0.16 \text{ e } \text{\AA}^{-3}$ , and the minimum value  $-0.15 \text{ e } \text{\AA}^{-3}$ . Programs used were *MULTAN82* (Main *et al.*, 1982), *ORTEP* (Johnson, 1965), *PLUTO78* (Motherwell & Clegg, 1978), and *SDP* (Frenz, 1978). Final positional and equivalent isotropic thermal parameters are shown in Table 1, and selected bond lengths, bond angles, and torsion

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	$x$	$y$	$z$	$B_{\text{eq}} (\text{\AA}^2)$
O1	0.3226 (2)	0.0848 (1)	0.51931 (8)	5.03 (3)
O2	0.3702 (3)	0.2617 (1)	0.5828 (1)	7.21 (5)
C1	0.2332 (5)	-0.2144 (2)	0.2918 (1)	6.32 (6)
C2	0.2303 (4)	-0.3049 (2)	0.3443 (2)	6.22 (6)
C3	0.1990 (4)	-0.2866 (2)	0.4319 (1)	5.38 (5)
C4	0.2163 (3)	-0.1772 (2)	0.4659 (1)	4.35 (4)
C5	0.2820 (3)	-0.0768 (2)	0.4102 (1)	4.35 (4)
C6	0.2519 (3)	0.0527 (2)	0.4368 (1)	4.27 (4)
C7	0.3526 (4)	0.1396 (2)	0.3799 (1)	5.05 (5)
C8	0.2742 (5)	0.1354 (2)	0.2927 (1)	6.66 (6)
C9	0.2899 (5)	0.0053 (3)	0.2628 (1)	6.80 (7)
C10	0.1969 (4)	-0.0868 (2)	0.3209 (1)	5.11 (5)
C11	0.3474 (3)	0.2502 (2)	0.4324 (2)	5.35 (5)
C12	0.3482 (4)	0.2059 (2)	0.5193 (2)	5.34 (5)
C13	0.3380 (5)	0.3651 (2)	0.4126 (2)	7.66 (7)
C14	-0.0172 (4)	-0.0702 (2)	0.3204 (2)	6.53 (6)
C15	0.1887 (3)	-0.1563 (2)	0.5571 (1)	5.03 (5)

Table 2. Bond distances ( $\text{\AA}$ ), bond angles ( $^\circ$ ), and selected torsion angles ( $^\circ$ )

O1—C6	1.466 (2)	C5—C10	1.565 (3)
O1—C12	1.360 (2)	C6—C7	1.513 (3)
O2—C12	1.207 (3)	C7—C8	1.513 (3)
C1—C2	1.316 (3)	C7—C11	1.493 (3)
C1—C10	1.517 (3)	C8—C9	1.529 (4)
C2—C3	1.444 (3)	C9—C10	1.539 (3)
C3—C4	1.340 (3)	C10—C14	1.537 (4)
C4—C5	1.508 (3)	C11—C12	1.484 (3)
C4—C15	1.500 (3)	C11—C13	1.320 (3)
C5—C6	1.519 (3)		
C6—O1—C12	106.7 (1)	C8—C7—C11	122.8 (2)
C2—C1—C10	121.0 (2)	C7—C8—C9	107.1 (2)
C1—C2—C3	121.5 (2)	C8—C9—C10	114.1 (2)
C2—C3—C4	121.0 (2)	C1—C10—C5	106.5 (2)
C3—C4—C5	117.2 (2)	C1—C10—C9	111.2 (2)
C3—C4—C15	122.0 (2)	C1—C10—C14	106.3 (2)
C5—C4—C15	120.6 (2)	C5—C10—C9	110.1 (2)
C4—C5—C6	119.4 (2)	C5—C10—C14	112.5 (2)
C4—C5—C10	111.9 (2)	C9—C10—C14	110.1 (2)
C6—C5—C10	105.8 (2)	C7—C11—C12	105.1 (2)
O1—C6—C5	116.0 (2)	C7—C11—C13	131.5 (2)
O1—C6—C7	103.3 (2)	C12—C11—C13	123.3 (2)
C5—C6—C7	111.6 (2)	O1—C12—O2	121.8 (2)
C6—C7—C8	111.6 (2)	O1—C12—C11	109.2 (2)
C6—C7—C11	99.9 (2)	O2—C12—C11	129.0 (2)
C12—O1—C6—C7	35.1 (2)	C6—C5—C10—C9	56.9 (3)
C6—O1—C12—C3	-15.8 (3)	O1—C6—C7—C11	-39.0 (2)
C10—C1—C2—C3	-2.2 (5)	C5—C6—C7—C8	64.3 (3)
C2—C1—C10—C5	34.6 (4)	C6—C7—C8—C9	-56.7 (3)
C1—C2—C3—C4	-16.1 (4)	C6—C7—C11—C12	29.7 (3)
C2—C3—C4—C5	-4.0 (4)	C7—C8—C9—C10	54.6 (3)
C3—C4—C5—C10	38.3 (3)	C8—C9—C10—C5	-56.9 (3)
C10—C5—C6—C7	-61.5 (2)	C7—C11—C12—O1	-9.8 (3)
C4—C5—C10—C1	-50.8 (3)	C13—C11—C12—O2	-12.8 (5)

angles are given in Table 2.\* The numbering scheme and thermal ellipsoids are illustrated in Fig. 1, and Fig. 2 is a stereoview of the unit cell.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53136 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

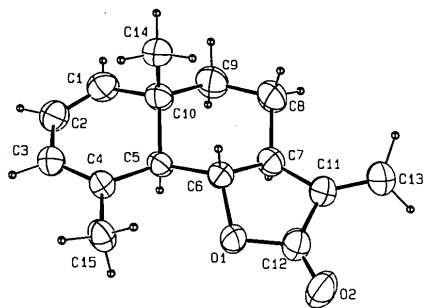


Fig. 1. The molecule of gazaniolide, with thermal ellipsoids drawn at the 40% probability level, and hydrogen atoms with arbitrary radius.

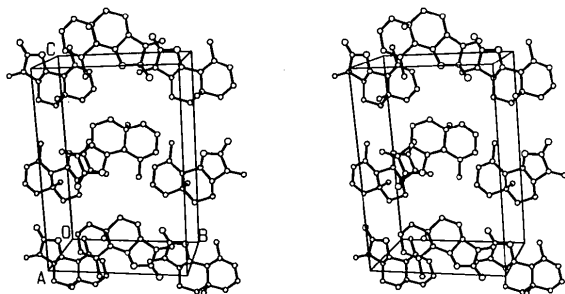


Fig. 2. Stereoview of the unit cell.

**Related literature.** Isolation of gazaniolide from the roots of *Rudbeckia subtomentosa*: Vasquez, Quijano, Fronczek, Macias, Urbatsch, Cox & Fischer (1990), occurrence of gazaniolide in *Gazania krebsiana*: Bohlmann & Zdero (1979), crystal structure of isodrimenin: Escobar & Wittke (1988), crystal structure of irazunolide: Hasbun, Calvo, Poveda,

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## *trans*-3,6-Dinonyl-1,2,4,5-tetroxane

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**Abstract.** *trans*-3,6-Dinonyl-1,2,4,5-tetroxane, C<sub>20</sub>H<sub>14</sub>O<sub>4</sub>, m.p. 340 K, *M<sub>r</sub>* = 172.3, triclinic, *P* $\bar{1}$ , *a* = 4.517 (2), *b* = 5.513 (1), *c* = 22.225(3) Å,  $\alpha$  = 89.46 (1),  $\beta$  = 84.07 (1),  $\gamma$  = 84.34 (1)°, *V* = 547.8 (3) Å<sup>3</sup>, *Z* = 1, *D<sub>x</sub>* = 1.04 Mg m<sup>-3</sup>,  $\lambda$ (Mo *K* $\alpha$ ) = 0.71069 Å,  $\mu$  = 0.066 mm<sup>-1</sup>, *F*(000) = 192, room

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