Table 1. *Fractional coordinates with standard deviations and equivalent isotropic thermal parameters for* MeCO-*ψ*[NH-CO]Val-NHMe

	x	y	z	B_{eq} (Å^2)
O(1)	0.5808 (4)	0.0228 (2)	0.1500 (2)	5.98 (6)
N(1)	0.1422 (5)	0.0595 (2)	0.1237 (1)	3.85 (5)
C(1)	0.1990 (8)	0.1134 (3)	0.0451 (2)	5.32 (7)
C(2)	0.3370 (5)	0.0170 (2)	0.1704 (2)	3.39 (5)
C(3)	0.2447 (7)	−0.0390 (2)	0.250	3.12 (7)
C(6)	0.3499 (8)	−0.1456 (2)	0.250	4.33 (8)
C(7)	0.264 (1)	−0.1993 (3)	0.1687 (3)	6.68 (9)
H(N1)	−0.0595	0.0488	0.1418	

Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsional angles* (°) *for* MeCO-*ψ*[NH-CO]Val-NHMe

N(1)—C(1)	1.445 (4)	C(2)—C(3)	1.514 (3)
N(1)—C(2)	1.322 (3)	C(3)—C(6)	1.530 (4)
O(1)—C(2)	1.230 (3)	C(6)—C(7)	1.511 (5)
C(1)—N(1)—C(2)	122.9 (2)	C(2)—C(3)—C(6)	111.9 (1)
O(1)—C(2)—N(1)	121.4 (3)	C(3)—C(6)—C(7)	111.1 (2)
O(1)—C(2)—C(3)	121.8 (2)	C(2)—C(3)—C(2^i)	108.9 (2)
N(1)—C(2)—C(3)	116.7 (3)	C(7)—C(6)—C(7^i)	112.7 (3)

	ψ	−110.4 (2)
C(2)—C(3)—C(6)—C(7)	χ^1/C(7)	−55.6 (4)
C(2)—C(3)—C(6)—C(7^i)	χ^1/C(7)′	178·1 (2)
C(1)—N(1)—C(2)—O(1)	ω	3 (4)

Final atomic parameters are given in Table 1.* Bond distances, bond angles and torsional angles are given in Table 2 (IUPAC–IUB Commission on Biochemical Nomeclature, 1970; IUPAC–IUB Joint Commission on Biochemical Nomenclature, 1984). Fig. 1 shows a thermal ellipsoid plot with the atomic numbering scheme. The conformation corresponds to a saddle point on the energy map (Stern, Chorev, Goodman & Hagler, 1983). Molecules are hydrogen bonded in such a way as to form a parallel *β*-sheet structure.

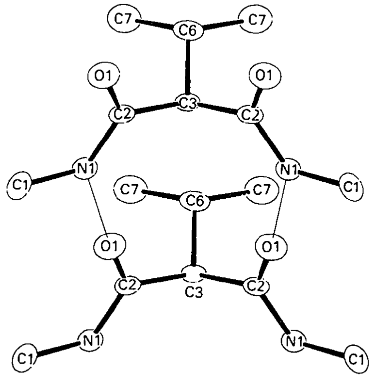

Fig. 1. *ORTEP* (Johnson, 1965) drawing of two associated MeCO-*ψ*[NH-CO]Val-NHMe molecules and interatomic distance (Å) associated with intermolecular hydrogen bond N—H···O [N(1)···O(1^i) = 2.803 (3); symmetry code: (i) $x - 1$, y, z].

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54437 (7 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 Guaianolide Derivative 9*α*-Thiophenoxy-11*βH*,13-dihydromicheliolide

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Abstract. $C_{21}H_{26}O_3S$, $M_r = 358.5$, monoclinic, $P2_1$, $a = 10.4847$ (10), $b = 5.4915$ (6), $c = 17.145$ (2) Å, $\beta = 96.366$ (10)°, $V = 981.0$ (3) Å^3, $Z = 2$, $D_x = 1.214$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 15.5$ cm^{-1}, $F(000) = 384$, $T = 297$ K, $R = 0.039$ for 1363 observations with $I > 1\sigma(I)$ (of 1590 unique data). The seven-membered ring is *trans* fused to the lactone ring and has a slightly distorted chair confor-

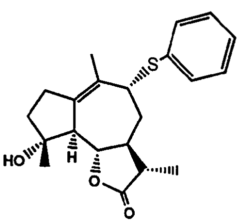
mation ($\Delta C_s = 6.3°$), with the local mirror bisecting the double bond. The lactone ring is in the envelope conformation with the O—CO bond lying across the local mirror, while the other five-membered ring is in the half-chair conformation with the C atom involved in the double bond on the twist axis. The hydroxy group forms a weak intermolecular hydrogen bond with the lactone carbonyl O atom related by a screw axis, having O···O distance 2.948 (4) Å and angle at H equal to 150 (5)°.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

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

	x	y	z	B_{eq}(Å^2)
S1	0.04179 (9)	0	0.63197 (5)	5.22 (2)
O1	0.0708 (2)	0.0345 (6)	0.9257 (1)	3.90 (5)
O2	−0.1078 (2)	0.0454 (6)	0.9850 (1)	5.36 (6)
O3	0.3007 (2)	0.3670 (5)	0.9160 (1)	4.69 (6)
C1	0.2864 (3)	−0.1091 (8)	0.7678 (2)	4.05 (8)
C2	0.4302 (3)	−0.063 (1)	0.7848 (2)	5.9 (1)
C3	0.4389 (3)	0.1622 (9)	0.8373 (2)	5.2 (1)
C4	0.3298 (3)	0.1324 (8)	0.8882 (2)	3.80 (8)
C5	0.2196 (3)	0.0374 (8)	0.8268 (2)	3.63 (7)
C6	0.1164 (3)	−0.1058 (7)	0.8618 (2)	3.21 (7)
C7	−0.0067 (3)	−0.1554 (8)	0.8087 (2)	3.52 (7)
C8	0.0065 (3)	−0.3487 (8)	0.7479 (2)	4.12 (8)
C9	0.0925 (3)	−0.2806 (8)	0.6853 (2)	4.35 (9)
C10	0.2348 (3)	−0.2506 (9)	0.7096 (2)	4.62 (9)
C11	−0.0973 (3)	−0.2105 (8)	0.8698 (2)	4.15 (8)
C12	−0.0507 (3)	−0.0342 (8)	0.9340 (2)	4.09 (8)
C13	−0.2405 (3)	−0.184 (1)	0.8429 (2)	6.9 (1)
C14	0.3155 (4)	−0.386 (1)	0.6557 (2)	6.9 (1)
C15	0.3638 (3)	−0.0409 (9)	0.9552 (2)	4.98 (9)
C16	−0.1211 (4)	−0.0595 (8)	0.5999 (2)	4.7 (1)
C17	−0.2150 (4)	0.089 (1)	0.6234 (2)	6.2 (1)
C18	−0.3440 (4)	0.046 (1)	0.5981 (3)	7.8 (1)
C19	−0.3767 (4)	−0.147 (1)	0.5508 (3)	8.3 (1)
C20	−0.2846 (5)	−0.294 (1)	0.5271 (3)	7.9 (1)
C21	−0.1576 (4)	−0.254 (1)	0.5504 (2)	6.5 (1)

Experimental. 9α-Thiophenoxy-11βH,13-dihydromicheliolide (1) was obtained from the BF_3-mediated rearrangement of dihydroparthenolide with thiophenol. Crystals formed from methanol–water solution, m.p. 437–439 K, were suitable; a tiny colorless needle with dimensions 0.025 × 0.05 × 0.35 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $20 > \theta > 10°$. The ω–2θ scans were designed for $I = 20\sigma(I)$, subject to maximum scan time = 300 s, scan rates varied from 0.18 to 3.30° min^{-1}. A quadrant of data having $2 < \theta < 60°$, $0 \leq h \leq 11$, $0 \leq k \leq 6$, $-19 \leq l \leq 19$ was measured and corrected for background, Lorentz and polarization effects. Absorption corrections were unnecessary. Three standard reflections (300, 020, 004) exhibited no significant variation in intensity and no decay correction was applied. A total of 1590 unique data was measured. The space group was determined by systematic absences 0k0 with k odd and the knowledge that the compound is chiral. The structure was solved by direct methods using *RANTAN* (Yao Jia-Xing, 1981), refined by full-matrix least squares based upon F, using 1363 data for which $I > 1\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors from Cromer & Waber (1974) and anomalous coefficients from Cromer (1974). Heavy-atom coordinates were refined with anisotropic thermal parameters. H-atom coordinates were located by ΔF synthesis and the hydroxy H-atom position was refined with an isotropic thermal parameter, while other H atoms were included as fixed contributors with C—H distance 0.95 Å and $B_{iso} = 1.3B_{eq}$ for the bonded C atom. Final $R = 0.039$ for 1363 observed data (0.066 for all 1590 data), $wR = 0.042$ and $S = 1.424$ for 230 variables. Maximum shift 0.09σ in the final cycle, maximum residual density 0.16, minimum −0.10 e Å^{-3} and extinction coefficient $g = 1.9\ (3) \times 10^{-6}$ where the factor $(1 + gI_c)^{-1}$ was applied to F_c. The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a perspective drawing showing the atom numbering and Fig. 2 illustrates the unit cell. Bond distances, angles and selected torsion angles are presented in Table 2.*



(1)

Related literature. The conformation of the title molecule is similar to that of micheliolide (Acosta, Fronczek & Fischer, 1991) except for that of the lactone ring, which is a half-chair in micheliolide. Bond lengths C11—C13 1.527 (5) and C12—O2 1.196 (4) Å of title molecule are similar to those of 3-oxograndolide [C11—C13 1.512 (5), C12—O2 1.200 (4) Å] (Rychlewska, 1985), to those of taurin [C11—C13 1.522 (5), 1.548 (5), C12—O2 1.202 (4), 1.203 (5) Å] (Calleri, Chiari & Viterbo, 1983) and to those of tauremisin [C11—C13 1.519 (4), C12—O2 1.202 (4) Å] (Tavanaiepour, Watson, Miski, Gage &

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* Lists of H-atom coordinates and thermal parameters, anisotropic thermal parameters, torsion angles and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54473 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

S1—C9	1.840 (4)	C6—C7	1.519 (4)
S1—C16	1.765 (4)	C7—C8	1.506 (6)
O1—C6	1.462 (4)	C7—C11	1.521 (5)
O1—C12	1.351 (4)	C8—C9	1.521 (5)
O2—C12	1.196 (4)	C9—C10	1.513 (5)
O3—C4	1.419 (5)	C10—C14	1.515 (6)
O3—H3O	0.86 (4)	C11—C12	1.507 (5)
C1—C2	1.525 (5)	C11—C13	1.527 (5)
C1—C5	1.522 (5)	C16—C17	1.372 (6)
C1—C10	1.331 (5)	C16—C21	1.391 (7)
C2—C3	1.524 (7)	C17—C18	1.393 (6)
C3—C4	1.522 (5)	C18—C19	1.359 (9)
C4—C5	1.563 (4)	C19—C20	1.355 (8)
C4—C15	1.505 (6)	C20—C21	1.365 (6)
C5—C6	1.515 (5)		

C9—S1—C16	102.5 (2)	C8—C7—C11	116.7 (3)
C6—O1—C12	108.8 (3)	C7—C8—C9	115.0 (3)
C4—O3—H3O	111 (4)	S1—C9—C8	113.3 (3)
C2—C1—C5	107.5 (3)	S1—C9—C10	105.5 (3)
C2—C1—C10	123.5 (3)	C8—C9—C10	118.4 (3)
C5—C1—C10	128.9 (3)	C1—C10—C9	125.2 (3)
C1—C2—C3	104.0 (3)	C1—C10—C14	122.4 (3)
C2—C3—C4	104.6 (3)	C9—C10—C14	112.3 (3)
O3—C4—C3	107.4 (3)	C7—C11—C12	101.4 (3)
O3—C4—C5	111.1 (3)	C7—C11—C13	116.2 (3)
O3—C4—C15	111.0 (3)	C12—C11—C13	112.9 (4)
C3—C4—C5	101.3 (3)	O1—C12—O2	122.0 (3)
C3—C4—C15	112.1 (3)	O1—C12—C11	109.6 (3)
C5—C4—C15	113.5 (3)	O2—C12—C11	128.4 (3)
C1—C5—C4	105.3 (2)	S1—C16—C17	120.0 (3)
C1—C5—C6	113.0 (3)	S1—C16—C21	121.6 (3)
C4—C5—C6	114.5 (3)	C17—C16—C21	118.4 (4)
O1—C6—C5	109.3 (3)	C16—C17—C18	121.0 (5)
O1—C6—C7	102.5 (2)	C17—C18—C19	119.2 (5)
C5—C6—C7	117.0 (3)	C18—C19—C20	120.3 (4)
C6—C7—C8	113.8 (3)	C19—C20—C21	121.3 (5)
C6—C7—C11	100.2 (2)	C16—C21—C20	119.8 (5)

C10—C1—C5—C6	48.5 (1)	O1—C6—C7—C11	-45.0 (1)
C2—C1—C5—C4	-9.4 (1)	C2—C1—C10—C9	-175.4 (1)
C5—C1—C10—C9	4.8 (2)	C5—C6—C7—C8	81.1 (1)
S1—C9—C10—C14	-99.8 (1)	C8—C9—C10—C14	133.1 (1)
C13—C11—C12—O2	37.4 (2)	C7—C8—C9—S1	-53.5 (1)
C9—S1—C16—C21	-60.7 (1)	C16—S1—C9—C8	-53.9 (1)
C1—C2—C3—C4	39.7 (1)	C2—C3—C4—C5	-45.0 (1)
C3—C4—C5—C1	33.6 (1)	C2—C1—C5—C4	-9.4 (1)
C5—C1—C2—C3	-18.4 (1)	C8—C9—C10—C1	-53.2 (2)
C7—C8—C9—C10	69.5 (1)	C6—C7—C8—C9	-72.4 (1)
C1—C5—C6—C7	-76.6 (1)	C6—C7—C11—C12	41.2 (1)
C7—C11—C12—O1	-25.1 (1)	C6—O1—C12—C11	-4.0 (1)
C12—O1—C6—C7	31.8 (1)		

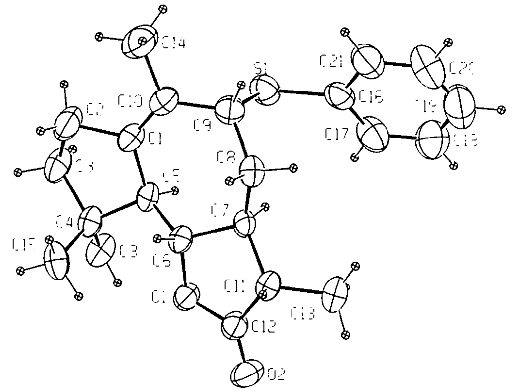

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as spheres of arbitrary radius.

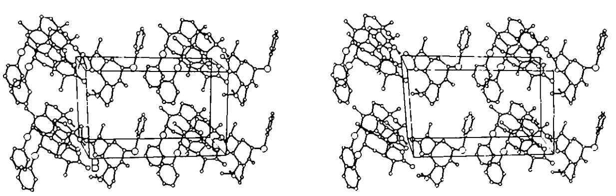

Fig. 2. Stereoview of the unit cell, viewed slightly oblique to the *b* axis. Only the H atom involved in hydrogen bonding is illustrated.

Mabry, 1987). The lactone exocyclic torsion angle O2—C12—C11—C13 and the torsion angle C11—C7—C6—O1 at the lactone seven-membered ring fusion bond are 37.4 (2) and −45.0 (1)° respectively, differing from those of 3-oxograndolide [42.3 (4) and −23.3 (3)° respectively] (Rychlewska, 1985). The C(sp^3)—S bond length, 1.840 (4) Å, and C(sp^2)—S, 1.765 (4) Å, of the title compound are similar to those of the *p*-bromobenzenethiol adduct of the $2\alpha,3\beta$-diacetoxyisolantolactone (Currie & Sim, 1973). The conformation associated with thiol addition can be described by the torsion angles C7—C8—C9—S1, −53.5 (1), and C8—C9—S1—C16, −53.9 (1)°. The torsion angle C9—S1—C16—C21 is −60.7 (1)°, and differs from that of the *p*-bromobenzenethiol adduct of the $2\alpha,3\beta$-diacetoxyisolantolactone [9 (1)°] (Currie & Sim, 1973).

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