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

$$B_{eq} = \tfrac{8}{3}\pi^2(U_{11} + U_{22} + U_{33}).$$

	x	y	z	B_{eq}
O	0.4784 (4)	0.0872 (1)	0.6692 (8)	5.4 (2)
N	1.2092 (5)	0.0344 (2)	0.9017 (9)	5.3 (2)
C	0.6000 (5)	0.1108 (2)	0.6025 (9)	3.7 (2)
C(11)	0.7606 (5)	0.0921 (2)	0.6801 (8)	3.4 (2)
C(12)	0.8966 (6)	0.1039 (2)	0.5519 (8)	3.9 (2)
C(13)	1.0466 (5)	0.0847 (2)	0.6199 (9)	3.8 (2)
C(14)	1.0618 (6)	0.0529 (2)	0.8277 (9)	3.7 (2)
C(15)	0.9223 (6)	0.0401 (2)	0.9560 (9)	4.1 (2)
C(16)	0.7762 (5)	0.0591 (2)	0.8816 (9)	3.7 (2)
C(20)	0.5165 (7)	0.2990 (2)	−0.0260 (1)	5.5 (3)
C(21)	0.5845 (5)	0.1588 (2)	0.4375 (8)	3.4 (2)
C(22)	0.4865 (6)	0.1548 (2)	0.2455 (1)	4.8 (3)
C(23)	0.4683 (7)	0.1992 (2)	0.0947 (1)	5.2 (3)
C(24)	0.5405 (6)	0.2502 (2)	0.1368 (9)	4.1 (2)
C(25)	0.6345 (6)	0.2538 (2)	0.3331 (1)	4.5 (2)
C(26)	0.6619 (6)	0.2088 (2)	0.4801 (9)	4.0 (2)

Figs. 1 and 2, respectively. The atomic parameters are listed in Table 1;* important bond lengths and bond angles are given in Table 2.

Related literature. 4-Aminobenzophenone (ABP) has a large second-harmonic generation efficiency (Frazier & Cockerham 1987) and also belongs to the monoclinic system with space group $P2_1$ (Su, Pan, Li, He & Huang, 1991). However, in ABP the carbonyl/aminophenyl dihedral angle of 6° is much smaller than the corresponding angle of 21.1° in 4-amino-4′-methylbenzophenone (AMBP) found here.

Table 2. *Intramolecular distances* (Å) *and angles* (°)

C—O	1.221 (5)	C(15)—C(16)	1.366 (6)
C—C(11)	1.479 (6)	C(21)—C(22)	1.363 (7)
C—C(21)	1.490 (6)	C(21)—C(26)	1.387 (6)
C(11)—C(12)	1.375 (6)	C(22)—C(23)	1.377 (7)
C(11)—C(16)	1.396 (6)	C(23)—C(24)	1.388 (7)
C(12)—C(13)	1.387 (6)	C(24)—C(20)	1.506 (7)
C(13)—C(14)	1.409 (7)	C(24)—C(25)	1.362 (6)
C(14)—N	1.372 (6)	C(25)—C(26)	1.384 (6)
C(14)—C(15)	1.405 (7)		

O—C—C(11)	121.1 (4)	C(14)—C(15)—C(16)	120.3 (4)
O—C—C(21)	118.8 (4)	C(15)—C(16)—C(11)	121.7 (4)
C(11)—C—C(21)	120.1 (4)	C(16)—C(11)—C(12)	118.2 (4)
C—C(11)—C(12)	121.7 (4)	C(21)—C(22)—C(23)	120.4 (5)
C—C(11)—C(16)	120.0 (4)	C(22)—C(23)—C(24)	122.2 (5)
C—C(21)—C(22)	119.8 (4)	C(23)—C(24)—C(20)	121.8 (5)
C—C(21)—C(26)	121.5 (4)	C(25)—C(24)—C(20)	121.8 (5)
C(11)—C(12)—C(13)	121.8 (4)	C(23)—C(24)—C(25)	116.4 (5)
C(12)—C(13)—C(14)	119.6 (4)	C(24)—C(25)—C(26)	122.5 (4)
N—C(14)—C(13)	120.8 (4)	C(25)—C(26)—C(21)	119.8 (4)
N—C(14)—C(15)	120.8 (4)	C(26)—C(21)—C(22)	118.6 (4)
C(13)—C(14)—C(15)	118.5 (4)		

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Structure of Hymenoratin

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Abstract. Hymenoratin, (3aα,4aβ,5β,6α,7aα,8α,9aα)-decahydro-5,6-dihydroxy-4a,8-dimethyl-3-methyleneazuleno[6,5-*b*]furan-2(3*H*)-one, $C_{15}H_{22}O_4$, $M_r = 266.34$, monoclinic, $P2_1$, $a = 13.525\ (7)$, $b = 7.549\ (4)$, $c = 13.646\ (7)$ Å, $\beta = 90.02\ (4)°$, $V = 1393.2\ (11)$ Å^3, $Z = 4$, $D_x = 1.270$ g cm^{-3}, λ(Mo $K\alpha$) = 0.70930 Å, $\mu = 0.849$ cm^{-1}, $F(000) = 576$, $T = 225$ K, $R = 0.046$ for 2838 unique observed reflections, $I \geq 3\sigma(I)$. The sesquiterpene lactone has its seven-membered ring *cis* fused to the α-methylene-

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Table 1. *Positional and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

	x	y	z	B* (Å^2)
O3′	0.0884 (2)	0.8205 (3)	−0.0062 (2)	3.18 (4)
O3	0.2230 (2)	1.4727 (3)	0.1691 (2)	3.73 (5)
O4′	0.1027 (1)	0.4147 (3)	−0.0035 (1)	2.53 (4)
O4	0.1838 (2)	1.1046 (3)	0.0656 (1)	2.99 (4)
O8′	0.1458 (2)	0.3052 (5)	−0.4807 (2)	4.43 (6)
O8	0.5051 (2)	0.610	0.2889 (2)	4.40 (5)
O12	0.5435 (2)	0.3961 (4)	0.1830 (2)	5.37 (6)
O12′	0.0690 (2)	0.0578 (5)	−0.5263 (2)	6.32 (7)
C1′	0.1871 (2)	0.6403 (4)	−0.2265 (2)	2.47 (5)
C1	0.3369 (2)	1.1050 (4)	0.2877 (2)	2.27 (5)
C2′	0.2010 (3)	0.7862 (5)	−0.1494 (2)	3.27 (6)
C2	0.2793 (2)	1.2800 (5)	0.3010 (2)	2.85 (6)
C3′	0.1586 (2)	0.7089 (4)	−0.0542 (2)	2.42 (5)
C3	0.2146 (2)	1.2995 (4)	0.2088 (2)	2.60 (5)
C4′	0.1116 (2)	0.5341 (4)	−0.0842 (2)	2.00 (5)
C4	0.2527 (2)	1.1542 (4)	0.1410 (2)	2.24 (5)
C5′	0.1790 (2)	0.4661 (4)	−0.1670 (2)	2.12 (5)
C5	0.2771 (2)	1.0004 (4)	0.2096 (2)	1.94 (5)
C6	0.3355 (2)	0.8520 (4)	0.1573 (2)	2.34 (5)
C6′	0.1342 (2)	0.3077 (4)	−0.2225 (2)	2.43 (5)
C7	0.4473 (2)	0.8389 (4)	0.1794 (2)	2.47 (5)
C7′	0.0796 (2)	0.3515 (5)	−0.3190 (2)	2.55 (5)
C8′	0.1451 (2)	0.4346 (5)	−0.4010 (2)	3.23 (7)
C8	0.4743 (2)	0.7984 (5)	0.2876 (2)	3.19 (6)
C9	0.3935 (2)	0.8135 (5)	0.3643 (2)	3.15 (6)
C9′	0.2516 (2)	0.4766 (6)	−0.3759 (2)	3.25 (6)
C10′	0.2639 (2)	0.6389 (5)	−0.3103 (2)	3.20 (6)
C10	0.3583 (2)	1.0052 (5)	0.3828 (2)	2.79 (6)
C11′	0.0401 (2)	0.1858 (6)	−0.3661 (2)	3.28 (6)
C11	0.4909 (2)	0.6842 (5)	0.1256 (2)	2.85 (6)
C12′	0.0831 (2)	0.1700 (7)	−0.4653 (2)	4.31 (8)
C12	0.5166 (2)	0.5452 (6)	0.1973 (3)	3.63 (7)
C13	0.1812 (2)	0.9218 (5)	0.2523 (2)	2.67 (5)
C13′	0.2799 (2)	0.4088 (5)	−0.1253 (2)	3.05 (6)
C14′	0.2586 (3)	0.8128 (6)	−0.3681 (3)	4.81 (8)
C14	0.4304 (3)	1.1090 (7)	0.4470 (2)	4.37 (8)
C15	0.5049 (3)	0.6605 (6)	0.0301 (2)	4.12 (8)
C15′	−0.0202 (3)	0.0662 (6)	−0.3325 (3)	4.28 (8)

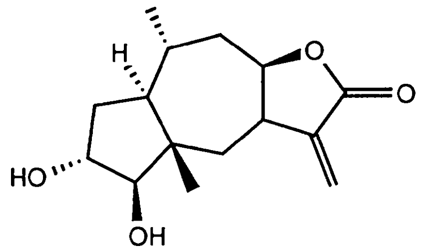
* $B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Principal bond distances* (Å) *and angles* (°)

O3′—C3′	1.429 (3)	C4—C5	1.528 (3)
O3—C3	1.420 (3)	C5′—C6′	1.539 (3)
O4′—C4′	1.429 (3)	C5′—C13′	1.541 (3)
O4—C4	1.436 (3)	C5—C6	1.545 (3)
O8′—C8′	1.461 (4)	C5—C13	1.542 (3)
O8′—C12′	1.343 (5)	C6—C7	1.545 (3)
O8—C8	1.480 (4)	C6′—C7′	1.545 (3)
O8—C12	1.353 (4)	C7—C8	1.551 (4)
O12—C12	1.199 (4)	C7—C11	1.500 (4)
O12′—C12′	1.202 (4)	C7′—C8′	1.560 (4)
C1′—C2′	1.535 (4)	C7′—C11′	1.504 (4)
C1′—C5′	1.549 (3)	C8′—C9′	1.514 (4)
C1′—C10′	1.544 (3)	C8—C9	1.518 (4)
C1—C2	1.545 (4)	C9—C10	1.544 (4)
C1—C5	1.553 (3)	C9′—C10′	1.526 (4)
C1—C10	1.528 (3)	C10′—C14′	1.534 (5)
C2′—C3′	1.535 (4)	C10—C14	1.527 (4)
C2—C3	1.538 (4)	C11′—C12′	1.478 (4)
C3′—C4′	1.521 (3)	C11′—C15′	1.300 (4)
C3—C4	1.525 (3)	C11—C12	1.476 (4)
C4′—C5′	1.540 (3)	C11—C15	1.329 (4)
C8′—O8′—C12′	112.8 (2)	C5—C6—C7	117.2 (2)
C8—O8—C12	111.7 (2)	C5′—C6′—C7′	116.2 (2)
C2′—C1′—C5′	105.0 (2)	C6—C7—C8	115.4 (2)
C2′—C1′—C10′	115.5 (2)	C6—C7—C11	109.8 (2)
C5′—C1′—C10′	115.5 (2)	C8—C7—C11	102.7 (2)
C2—C1—C5	104.6 (2)	C6′—C7′—C8′	115.2 (2)
C2—C1—C10	114.7 (2)	C6′—C7′—C11′	110.9 (2)
C5—C1—C10	115.5 (2)	C8′—C7′—C11′	103.2 (2)
C1′—C2′—C3′	105.2 (2)	O8′—C8′—C7′	105.6 (2)
C1—C2—C3	105.8 (2)	O8′—C8′—C9′	107.6 (2)
O3′—C3′—C2′	114.3 (2)	C7′—C8′—C9′	117.5 (2)
O3′—C3′—C4′	110.9 (2)	O8—C8—C7	105.5 (2)
C2′—C3′—C4′	104.9 (2)	O8—C8—C9	105.4 (3)
O3—C3—C2	110.8 (2)	C7—C8—C9	118.2 (2)
O3—C3—C4	113.8 (2)	C8—C9—C10	113.9 (2)
C2—C3—C4	103.7 (2)	C8′—C9′—C10′	113.9 (2)
O4′—C4′—C3′	112.0 (2)	C1′—C10′—C9′	111.5 (2)
O4′—C4′—C5′	113.9 (2)	C1′—C10′—C14′	110.1 (2)
C3′—C4′—C5′	103.9 (2)	C9′—C10′—C14′	112.4 (2)
O4—C4—C3	113.8 (2)	C1—C10—C9	112.4 (2)
O4—C4—C5	112.4 (2)	C1—C10—C14	110.8 (2)
C3—C4—C5	104.3 (2)	C9—C10—C14	112.2 (2)
C1′—C5—C4′	98.3 (2)	C7′—C11′—C12′	108.6 (3)
C1′—C5′—C6′	115.4 (2)	C7′—C11′—C15′	130.4 (2)
C1′—C5′—C13′	111.7 (2)	C12′—C11′—C15′	121.0 (3)
C4′—C5′—C6′	112.8 (2)	C7—C11—C12	108.8 (2)
C4′—C5′—C13′	110.3 (2)	C7—C11—C15	129.8 (3)
C6′—C5′—C13′	108.2 (2)	C12—C11—C15	121.4 (3)
C1—C5—C4	98.5 (2)	O8′—C12′—O12′	121.8 (3)
C1—C5—C6	114.8 (2)	O8′—C12′—C11′	109.3 (3)
C1—C5—C13	112.0 (2)	O12′—C12′—C11′	128.9 (3)
C4—C5—C6	112.2 (2)	O8—C12—O12	121.8 (3)
C4—C5—C13	110.0 (2)	O8—C12—C11	109.1 (3)
C6—C5—C13	109.0 (2)	O12—C12—C11	129.1 (3)

γ-lactone ring. Conformational detail of the two crystallographically independent molecules differs only slightly. The cycloheptane ring conformation is a slightly twisted boat; ΔC_2 values are 4.5 and 5.1. Individual torsion angles in the cycloheptane ring agree within ± 8°. The α-methylene-γ-lactone ring adopts an envelope conformation with C(7) at the flap of the unprimed molecule, but a flatter conformation with C(8) out-of-plane in the primed molecule. All hydroxyl donors participate in the hydrogen-bonding scheme, but not all possible acceptors. A hydrogen bond between the two molecules of the asymmetric unit is found [O3′···O4, 2.688 (3) Å]. Symmetry related molecules are linked [O4′···O3′, 2.684 (2) Å] and molecules translated along the *b* axis are linked [O3···O4′, 2.894 (3) Å; O4′···O4, 2.751 (3) Å]. One hydroxyl thus acts as a double acceptor (O4) and one (O3) does not accept any hydrogen bonds. Likewise, the lactone carbonyl is not hydrogen bonded. An additional close intramolecular contact [O3′···O4′, 3.070 (3) Å] is present. It is the disparate hydrogen-bonding interactions in which each molecule participates which principally distinguishes one from the other.

Experimental. The material, (I), was obtained from a methylene chloride extract of *Hymenoxys rusbyi* and crystallized from a mixture of cyclohexane, ethyl acetate and methanol. The crystal used for data collection was block shaped with approximate dimensions of 0.50 × 0.40 × 0.40 mm and was cemented to a glass fiber with epoxy resin. Cell dimensions were determined from the setting angles of 25 reflections with ($30 \leq 2\theta \leq 35°$) as collected on an Enraf–Nonius CAD-4 diffractometer equipped with graphite-monochromated molybdenum radiation. Variable speed ω–2θ scans (2.5 to 13.2° min^{-1}), extended by 25% on each side to collect background counts, were used for data collection in the range $2° \leq 2\theta \leq 56°$; h (0,16), k (0,9), l (−16,16). Data were corrected for Lorentz and polarization effects but not for absorption. Symmetry equivalent reflections within the quadrant $(+h, +k, \pm l)$ collected were averaged; $R_{\text{int}} = 0.020$. Of 3742 data collected, 3604 were unique, 359 were marked weak in a prescan, and 2838 were considered observed $[I \geq 3\sigma(I)]$ and used in the refinement. Intensity standards were monitored at the beginning, end and every three hours of exposure time: these showed no systematic alterations (±1.0% maximum).



(I)

Structure was solved with *SHELXS* (Sheldrick, 1985). The y coordinate of atom O(8) was held fixed to define the origin. Refinement by full-matrix least squares minimized the function $\sum w(|F_o| - |F_c|)^2$ where the weights, w, eventually were assigned as $4F_o^2/\sigma^2(I)$ with $\sigma^2(I)$ defined as $[\sigma^2(I_c) + (0.05F_o^2)^2]$. Non-H atoms were refined with anisotropic displacement parameters. H-atom positions were suggested from difference Fourier maps. With suggested positions as a guide, H atoms attached to C atoms were added to the refinement in fixed locations with fixed isotropic thermal values assigned as 1.3 times the value of $B_{\text{iso}}(\text{C})$. Hydroxyl hydrogen positions were allowed to refine. The refinement converged (max Δ/σ 0.19) to values of the standard agreement factors $R = 0.045$, $wR = 0.059$, goodness of fit = 1.575 with 354 variables. A final difference Fourier map showed maximum features of $\pm 0.263 \text{ e Å}^{-3}$. Values of the neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) as incorporated in the Enraf–Nonius *SDP* suite (Frenz, 1987) which was the source of all programs. Atomic coordinates are presented in Table 1;* bond distances and angles are listed in Table 2. A view of one of the crystallographically independent molecules is provided as Fig. 1.

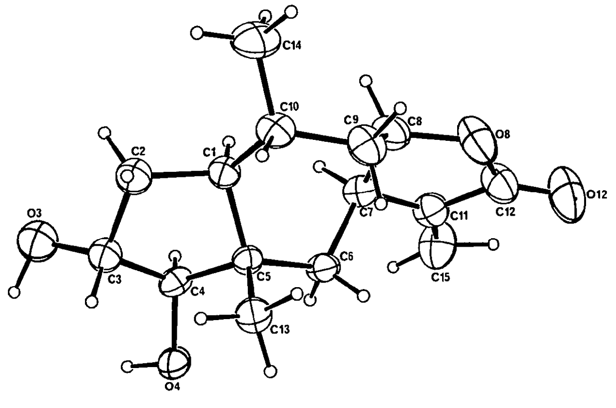


Fig. 1. *ORTEP* (Johnson, 1976) drawing of one of the two crystallographically independent molecules in the crystal structure of hymenoratin. Principal ellipsoids are drawn at the 50% probability level; H atoms as small spheres of arbitrary size. The atom-numbering scheme was chosen to correspond to schemes used for sesquiterpene lactones by previous authors. This scheme does not correlate with numbers used in IUPAC nomenclature.

Relelated literature. This sesquiterpene lactone crystallized pseudoisomorphously to geigerinin (Bloszyk, Dudek, Kosturkiewicz, Rychlewska, Daniewski, Gumulka, Nawrot, Budesinsky, Vasickova & Holub, 1989), a diastereoisomer which differs in stereochemistry only at C8. Three of the four hydrogen-bonding interactions in which the hydroxyls of these two structures participate involve the same pairs of atoms. Numerous sesquiterpene lactone structures have been reported in the literature; some of the most recent include helenalin and mexicanin I (Watson & Kashyap, 1990) and paucin (Sun, Chan, Baures & Eggleston, 1991). Review articles on sesquiterpene lactones are available (Seaman, 1982).

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* Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54542 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0527]