Table 2. *Selected bond lengths* (Å) *and angles* (°)

Fe(1)–Cl(1)	2·190 (2)	Fe(1)–Cl(2)	2·191 (2)
Fe(1)–Cl(3)	2·185 (2)	Fe(1)–Cl(4)	2·170 (2)
P(1)–N(1)	1·577 (4)	P(1)–C(11)	1·798 (5)
P(1)–C(21)	1·799 (5)	P(1)–C(31)	1·797 (5)
P(2)–N(1)	1·580 (4)	P(2)–C(41)	1·803 (5)
P(2)–C(51)	1·801 (5)	P(2)–C(61)	1·795 (5)
Cl(1)–Fe(1)–Cl(3)	108·0 (1)	Cl(1)–Fe(1)–Cl(2)	109·3 (1)
Cl(1)–Fe(1)–Cl(4)	109·7 (1)	Cl(2)–Fe(1)–Cl(3)	109·9 (1)
Cl(3)–Fe(1)–Cl(4)	111·8 (1)	Cl(2)–Fe(1)–Cl(4)	108·2 (1)
N(1)–P(1)–C(11)	106·9 (2)	N(1)–P(1)–C(21)	115·8 (2)
C(11)–P(1)–C(21)	107·6 (2)	N(1)–P(1)–C(31)	112·0 (2)
C(11)–P(1)–C(31)	107·7 (2)	C(21)–P(1)–C(31)	106·6 (2)
N(1)–P(2)–C(41)	112·3 (2)	N(1)–P(2)–C(51)	113·8 (2)
C(41)–P(2)–C(51)	108·5 (2)	N(1)–P(2)–C(61)	107·9 (2)
C(41)–P(2)–C(61)	106·5 (2)	C(51)–P(2)–C(61)	107·5 (2)
P(1)–N(1)–P(2)	141·8 (3)		

Table 1, and selected bond lengths and angles in Table 2.* Fig. 1 shows the atom-numbering scheme.

Related literature. Other salts of $FeCl_4^-$: Zaslow & Rundle (1957), Kistenmacher & Stucky (1968), Richards & Gregory (1965), Müller, Conradi, Demant & Dehnicke (1984), Bottomley, Carter, Engelhardt, Lincoln, Patrick & White (1984).

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* Lists of structure factors, H-atom coordinates, bond lengths and angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43072 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Helenalin, a Pseudoguaianolide from *Helenium amarum*

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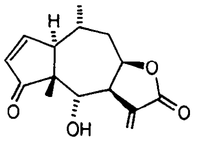
Abstract. 6α,8β-Dihydroxy-4-oxoambrosa-2,11(13)-dien-12-oic acid 12,8-lactone, $C_{15}H_{18}O_4$, $M_r = 262{\cdot}3$, monoclinic, $P2_1$, $a = 6{\cdot}1240$ (3), $b = 8{\cdot}2022$ (11), $c = 13{\cdot}8176$ (5) Å, $\beta = 98{\cdot}964$ (4)°, $V = 685{\cdot}6$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}271$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 7{\cdot}14$ cm^{-1}, $F(000) = 280$, $T = 299$ K, $R = 0{\cdot}036$ for 1394 observations (of 1528 unique data). Crystals of helenalin were isolated from *Helenium amarum* (Asteraceae) which was collected in East Baton Rouge Parish, Louisiana. The molecular structure agrees well with the previous determination of bromohelenalin [Ul-Haque & Caughlan (1969). *J. Chem. Soc. B*, pp. 956–960], including the molecular conformation. These two molecules exhibit a mean deviation of 4·1° over the 27 endocyclic torsion angles, with the largest single difference being 19·4°, for C(10)—C(1)—C(5)—C(6). The seven-membered ring in both has a distorted C_2 twist-chair conformation, the pseudodiad passing through C(10), with asymmetry parameter 13·6° for helenalin. Molecules are linked in the crystal in chains along the symmetry axis by hydrogen bonds involving the hydroxyl group and the carbonyl at C(4). The O···O distance is 2·728 (3) Å.

Experimental. The title compound (I) was obtained as colorless needles, dimensions 0·16 × 0·20 × 0·68 mm.



(I)

Space group from systematic absences 0*k*0 with *k* odd and chirality of material. Enraf–Nonius CAD-4 diffractometer with graphite monochromator and Cu $K\alpha$ radiation. Cell dimensions from setting angles of 25 reflections having $70 < 2\theta < 72°$. Data collection by ω–2θ scans designed for $I = 50\sigma(I)$, subject to 180 s maximum scan time. Scan rates varied 0·33–4·0° min^{-1}. Reflections having $2 < \theta < 75°$, $0 \le h \le 7$,

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Table 1. *Coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = \frac{1}{3}(a^2a^{*2}B_{11} + B_{22} + c^2c^{*2}B_{33} + aca^*c^*B_{13}\cos\beta)$.

	x	y	z	B_{eq}
O(1)	0·3280 (3)	0·04473	0·4819 (1)	5·34 (4)
O(2)	0·2058 (3)	0·5809 (3)	0·1206 (1)	5·59 (4)
O(3)	−0·0633 (4)	0·7505 (3)	0·1485 (1)	6·50 (5)
O(4)	0·5805 (2)	0·3058 (2)	0·3804 (1)	4·09 (3)
C(1)	0·3925 (3)	0·0937 (3)	0·2305 (1)	3·10 (4)
C(2)	0·4703 (3)	−0·0756 (3)	0·2603 (2)	3·83 (4)
C(3)	0·4560 (4)	−0·1102 (3)	0·3537 (2)	4·15 (5)
C(4)	0·3540 (3)	0·0267 (3)	0·3964 (2)	3·66 (4)
C(5)	0·2723 (3)	0·1489 (3)	0·3153 (1)	2·88 (3)
C(6)	0·3472 (3)	0·3182 (3)	0·3501 (1)	2·98 (3)
C(7)	0·3016 (3)	0·4571 (3)	0·2745 (1)	3·07 (4)
C(8)	0·3074 (4)	0·4296 (3)	0·1641 (1)	4·02 (4)
C(9)	0·1756 (4)	0·2913 (4)	0·1100 (1)	4·53 (5)
C(10)	0·2735 (4)	0·1181 (3)	0·1256 (1)	4·14 (5)
C(11)	0·0942 (3)	0·5555 (3)	0·2717 (2)	3·53 (4)
C(12)	0·0602 (4)	0·6409 (4)	0·1760 (2)	4·69 (5)
C(13)	−0·0338 (4)	0·5810 (4)	0·3379 (2)	5·54 (6)
C(14)	0·4295 (5)	0·0813 (5)	0·0530 (2)	7·08 (8)
C(15)	0·0196 (3)	0·1281 (3)	0·2982 (2)	3·97 (4)

$0 \leq k \leq 10$, $-17 \leq l \leq 17$ measured, corrected for background, Lorentz and polarization effects. Absorption correction by ψ scans, minimum relative transmission 94·43%. Standard reflections (300, 040, 005), ±1·7% max. variation. Redundant $0kl$ and $0k\bar{l}$ data averaged, $R_{int} = 0·016$. Structure solved using *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refined by full-matrix least squares based on F using data for which $I > 3\sigma(I)$, 134 unobserved reflections, $w = 4F_o^2[\sigma^2(I) + (0·02F_o^2)^2]^{-1}$, with Enraf–Nonius *SDP* (Frenz & Okaya, 1980). Non-H atoms anisotropic; H atoms located by ΔF and refined isotropically, one H atom on C(14) fixed, $B = 8·0$ Å^2. Atomic scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Final $R = 0·036$, $wR = 0·048$, $S = 2·372$ for 240 variables, extinction coefficient (Darwin, 1922) $g = 1·36\,(6) \times 10^{-5}$ ($|F_c| = |F_o|[1 + gIc]$), max. shift in final cycle $0·13\sigma$, max. residual density 0·14, min. −0·17 e Å^{-3}. Refinement of enantiomorph inconclusive, and the absolute configuration assumed to correspond to that of plenolin (Lee, Ibuka, McPhail, Onan, Geissman & Waddell, 1974). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond distances and angles in Table 2, and endocyclic torsion angles describing the conformation in Table 3. Fig. 1 shows the atom-numbering scheme.

Related literature. Crystal structure of helenalin oxide (McPhail & Onan, 1975); structure of bromohelenalin

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

O(1)–C(4)	1·226 (3)	C(5)–C(6)	1·517 (3)
O(2)–C(8)	1·474 (3)	C(5)–C(15)	1·538 (3)
O(2)–C(12)	1·355 (3)	C(6)–C(7)	1·542 (3)
O(3)–C(12)	1·197 (4)	C(7)–C(8)	1·547 (4)
O(4)–C(6)	1·429 (3)	C(7)–C(11)	1·500 (3)
C(1)–C(2)	1·505 (3)	C(8)–C(9)	1·520 (4)
C(1)–C(5)	1·545 (3)	C(9)–C(10)	1·544 (4)
C(1)–C(10)	1·530 (3)	C(10)–C(14)	1·520 (5)
C(2)–C(3)	1·338 (3)	C(11)–C(12)	1·483 (3)
C(3)–C(4)	1·454 (4)	C(11)–C(13)	1·311 (4)
C(4)–C(5)	1·529 (3)	C–H range	0·86 (4)–1·19 (5)
C(8)–O(2)–C(12)	110·4 (2)	C(5)–C(6)–C(7)	116·6 (3)
C(2)–C(1)–C(5)	103·4 (2)	C(6)–C(7)–C(8)	122·1 (3)
C(2)–C(1)–C(10)	117·6 (2)	C(6)–C(7)–C(11)	118·5 (3)
C(5)–C(1)–C(10)	117·8 (3)	C(8)–C(7)–C(11)	101·7 (3)
C(1)–C(2)–C(3)	113·2 (3)	O(2)–C(8)–C(7)	101·9 (2)
C(2)–C(3)–C(4)	108·9 (3)	O(2)–C(8)–C(9)	105·6 (3)
O(1)–C(4)–C(3)	127·8 (2)	C(7)–C(8)–C(9)	120·1 (3)
O(1)–C(4)–C(5)	123·4 (2)	C(8)–C(9)–C(10)	116·9 (3)
C(3)–C(4)–C(5)	108·7 (3)	C(1)–C(10)–C(9)	111·7 (3)
C(1)–C(5)–C(4)	102·9 (3)	C(1)–C(10)–C(14)	110·1 (3)
C(1)–C(5)–C(6)	110·6 (3)	C(9)–C(10)–C(14)	111·1 (3)
C(1)–C(5)–C(15)	116·0 (3)	C(7)–C(11)–C(12)	106·1 (3)
C(4)–C(5)–C(6)	108·6 (3)	C(7)–C(11)–C(13)	131·6 (3)
C(4)–C(5)–C(15)	104·3 (3)	C(12)–C(11)–C(13)	122·1 (3)
C(6)–C(5)–C(15)	113·4 (3)	O(2)–C(12)–O(3)	122·2 (2)
O(4)–C(6)–C(5)	105·5 (2)	O(2)–C(12)–C(11)	108·6 (2)
O(4)–C(6)–C(7)	108·6 (2)	O(3)–C(12)–C(11)	129·1 (2)

Table 3. *Selected torsion angles* (°)

C(1)–C(2)–C(3)–C(4)	3·6 (5)	C(7)–C(8)–C(9)–C(10)	−77·8 (5)
C(2)–C(3)–C(4)–C(5)	7·7 (5)	C(8)–C(9)–C(10)–C(1)	35·5 (5)
C(3)–C(4)–C(5)–C(1)	−15·1 (5)	C(9)–C(10)–C(1)–C(5)	47·4 (5)
C(4)–C(5)–C(1)–C(2)	16·1 (4)	C(7)–C(8)–O(2)–C(12)	−29·3 (4)
C(5)–C(1)–C(2)–C(3)	−13·1 (4)	C(8)–O(2)–C(12)–C(11)	13·2 (4)
C(10)–C(1)–C(5)–C(6)	−96·4 (4)	O(2)–C(12)–C(11)–C(7)	9·3 (4)
C(1)–C(5)–C(6)–C(7)	61·6 (5)	C(12)–C(11)–C(7)–C(8)	−26·1 (5)
C(5)–C(6)–C(7)–C(8)	−33·6 (5)	C(11)–C(7)–C(8)–O(2)	32·6 (4)
C(6)–C(7)–C(8)–C(9)	51·4 (5)	C(13)–C(11)–C(12)–O(3)	8·0 (4)

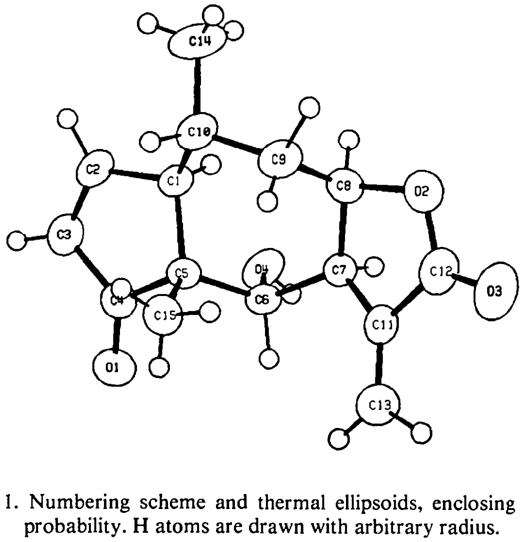

Fig. 1. Numbering scheme and thermal ellipsoids, enclosing 40% probability. H atoms are drawn with arbitrary radius.

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43371 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Ul-Haque & Caughlan, 1969); structure of plenolin (11,13-dihydrohelenalin) as *p*-iodobenzoate (Lee, Ibuka, McPhail, Onan, Geissman & Waddell, 1974); structure of autumnolide (Von Dreele, Petit, Cragg & Ode, 1975); structure of mexicanin-E as monobromide (Ul-Haque & Caughlan, 1967); various plant sources (Fischer, Olivier & Fischer, 1979); conformational asymmetry parameters (Duax & Norton, 1975).

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Structure of a Local Anaesthetic: Dyclonine Hydrochloride*

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Abstract. 3-Piperidino-4′-butoxypropiophenone hydrochloride, $C_{18}H_{28}NO_2^+.Cl^-$, $M_r = 325{\cdot}9$, monoclinic, $P2_1/a$, $a = 9{\cdot}1084\ (3)$, $b = 14{\cdot}3242\ (7)$, $c = 14{\cdot}8573\ (5)$ Å, $\beta = 106{\cdot}523\ (3)°$, $V = 1858{\cdot}39\ (4)$ Å^3, $T = 295$ K, $Z = 4$, $D_x = 1{\cdot}165\ (2)$, $D_m = 1{\cdot}16\ (3)$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 18{\cdot}8$ cm^{-1}, $F(000) = 704$, final $R = 0{\cdot}0346$, $wR = 0{\cdot}0327$ for 2720 significant reflections ($I_{\rm net} \geq 2{\cdot}5\sigma$). The piperidine ring is in a distortion-free chair conformation. The phenyl ring makes an angle of 29·9 (2)° with the best plane through the piperidine ring. The C—C—C—C—N group linking the benzene and the piperidine ring is in *trans–trans* conformation. The N atom of the piperidine ring is hydrogen-bonded to the Cl atom, N···Cl 3·062 (2) Å, N—H···Cl 170·2 (2)°.

Experimental. The title compound is a local anaesthetic. The X-ray analysis of the compound was undertaken as a part of a project on the crystal structure and conformation of local anaesthetics. The sample was obtained from Sigma Chemical Co. Colourless needle-shaped crystals, 0·3 × 0·25 × 0·15 mm, from a mixture of methanol + ether. D_m by flotation. Preliminary data from oscillation and Weissenberg photographs, cell constants refined using 70 reflections, $110 \leq 2\theta \leq 120°$ and Cu $K\alpha$ ($\lambda = 1{\cdot}54051$ Å), $\theta/2\theta$ scan with line profile analysis (Grant & Gabe, 1978), Picker four-circle automatic diffractometer, graphite-monochromated Cu $K\alpha$ radiation, no absorption or extinction corrections, data corrected for polarization and Lorentz effects (Le Page, Gabe & Calvert, 1979), 3309 reflections measured, $2\theta_{\max} = 120°$. $-10 \leq h \leq 9$, $0 \leq k \leq 16$, $0 \leq l \leq 16$, 2896 independent reflections, 2760 observed with $I \geq 2{\cdot}5\sigma(I)$. Agreement between equivalent reflections 0·7% on intensity. Three standard reflections monitored, variation <2%. Structure solution by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Initial $R = 0{\cdot}35$, after few cycles of refinement $R = 0{\cdot}15$. Hydrogen atoms from $\Delta\rho$ synthesis, full-matrix least-squares refinement on $|F|$, non-hydrogen atoms aniso-

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* DCB Contribution No. 690.

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