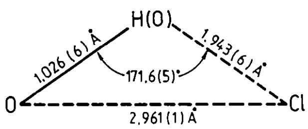
The packing in the structure is shown in Fig. 2. The 2-[(hydroxyimino)(phenylthio)methyl]-1-methylpyridinium cations and chloride anions are linked through a hydrogen bridge, O—H(O)···Cl, with the geometry illustrated in (2).



(2)

The existence of such a bond between halide anions and the oxygen of the oxime group has been postulated earlier in 2-(hydroxyiminomethyl)-1-methylpyridinium (2-PAM) iodide (Carlström, 1966) and in 1-benzyl-2-(hydroxyiminomethyl)pyridinium bromide (Van Havere, Lenstra & Geise, 1982), and confirmed recently in 2-(hydroxyiminomethyl)-1-methylpyridinium chloride (Van Havere, Lenstra, Geise, Van den Berg & Benschop, 1982).

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Absolute Structure of (+)-8α-Acetoxy-12-(4-bromobenzoyloxy)-13,14,15,16-tetranorlabdane,* $C_{25}H_{35}BrO_4$ (I), and Structure of (−)-8α,12-Dihydroxy-13,14,15,16-tetranor-9-epilabdane,† $C_{16}H_{30}O_2$ (II)

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Abstract. (I): $M_r = 479{\cdot}5$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}1383$ (7), $b = 17{\cdot}093$ (3), $c = 22{\cdot}912$ (5) Å, $V = 2404{\cdot}0$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}325$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}717$ mm^{-1}, $F(000) = 1008$, room temperature, $R = 8{\cdot}5\%$ for 1828 observed reflections (mostly Friedel pairs), $[\alpha]_D^{20^\circ C} = +2{\cdot}1^\circ$ (1·38% in $CHCl_3$), m.p. 361–363·5 K. (II): $M_r = 254{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}235$ (1), $b = 11{\cdot}931$ (3), $c = 17{\cdot}064$ (4) Å, $V = 1473{\cdot}0$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}147$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}068$ mm^{-1}, $F(000) = 568$, room temperature, $R = 3{\cdot}7\%$ for 624 observed reflections (mostly Friedel pairs), $[\alpha]_D^{20^\circ C} = -13{\cdot}6^\circ$ (1·54% in $CHCl_3$), m.p. 383–384 K. The absolute configuration for chiral centres of (I) was confirmed by least-squares refinement. For the two structures, the six-membered rings are *trans*-fused and both are in the chair conformation. There are no unusual bond distances or angles.

* (+)-12-(4-Bromobenzoyloxy)-13,14,15,16-tetranorlabdan-8α-yl acetate.

† (−)-13,14,15,16-Tetranor-9β-labdane-8α,12-diol.

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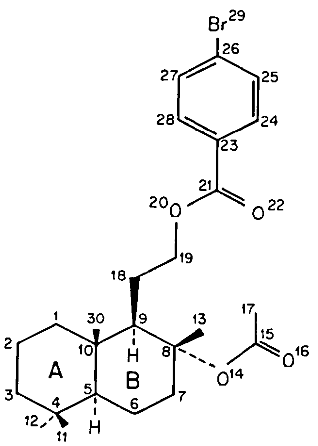
Table 1. *Summary of crystal data, intensity measurement and structure refinement*

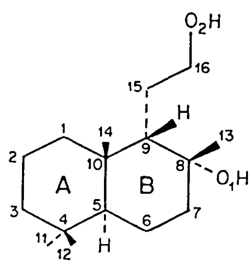
	(I)	(II)
Crystal size (mm)	0·18 × 0·23 × 0·32	0·13 × 0·28 × 0·28
Unit-cell determination	Least-squares fit from	
	26 reflections (19° ≤ 2θ ≤ 23°)	28 reflections (21° ≤ 2θ ≤ 30°)
$(\sin\theta/\lambda)_{max}$(Å^{-1})	0·53	0·55
h,k,l range	0–6, 0–17, 0–24	0–7, 0–13, 0–18
	(and all anti-reflections of these)	
No. of standard reflections (variation)	3 (1·8%)	3 (1·4%)
Number of Friedel pairs measured	1724	1198
Criterion for observed reflections	$\|F\| > 3\sigma(F)$ and $\|F\| > 8$	$\|F\| > 3\sigma(F)$ and $\|F\| > 7$
Number of observed reflections	1828	624
Refinement (on *F*)	Two blocks	Full-matrix
Number of parameters	273	164 for non-H atoms 92 for H atoms
Weighting scheme	$w(F)=(\|F\|/48)^2$ for $\|F\| \le 48$ and $(48/\|F\|)^2$ for $\|F\| > 48$	$w(F)=\exp[18(\sin\theta/\lambda)^2]$*
H atoms	Calculated	Refined
	U_{iso} fixed to 0·05 Å^2	
Max. and min. ratio of |shift| to error		
for non-H atoms	0·58, 0·16	0·03, 0·008
for H atoms	—	0·95, 0·10
Max. and min. $\Delta\rho$ (e Å^{-3})	0·62, −0·72	0·25, −0·32
S	1·28	2·66
R, *wR* (%)	8·5, 5·6	3·7, 3·8
Absolute-structure parameter *x*†	−0·02 (3)	—

* Dunitz & Seiler (1973).
† Flack (1983).

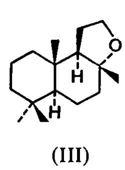
Introduction. In a previous paper (Bernardinelli, Dunand, Flack, Yvon, Giersch & Ohloff, 1984) we showed that the decalin skeleton of an intermediate in the synthesis of (+)-isoambrox possesses an ideal chair conformation. We now present the X-ray diffraction analysis of (I) and (II), two intermediates in the synthesis of (−)-Ambrox® (III) (Ohloff, 1982), and the diastereoisomeric ether (IV); all possess the same conformation despite the change in odour strength and quality of the three tricyclic ethers (Ohloff, 1985). These compounds are derived, with retention of configuration, from (+)-norambreinolide, whose absolute configuration is known (Klyne & Buckingham, 1978).



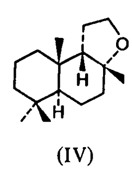
(I)



(II)



(III)



(IV)

Experimental. Experimental data and structure refinement are summarized in Table 1. D_m not determined; Philips PW 1100 diffractometer, graphite-monochromated Mo $K\alpha$; $\omega/2\theta$ scans; Lorentz–polarization correction; no absorption correction; systematic absences: $h00$: $h = 2n+1$, $0k0$: $k = 2n+1$, $00l$: $l = 2n+1$; structures solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); atomic scattering factors and anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974); no secondary-extinction correction; all calculations performed with a local version of *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *ORTEP*II (Johnson, 1976).

Discussion. Final positional parameters and equivalent isotropic temperature factors are given in Table 2.* Bond distances and torsion angles around the rings *A* and *B* are to be found in Table 3. In both structures, the rings *A* and *B* are in the chair conformation, and are *trans*-fused. Hence the substituents at C(8) and the side chain at C(9) are attached in ideal equatorial positions for (I) and in axial positions for (II).

(I): Since the absolute-structure parameter *x* (Flack, 1983) refined to −0·017 (27), the chirality of the crystal and of the coordinates are the same. They are referred to right-handed axes and the correct absolute configuration of the molecule is shown in Fig. 1. The

* Lists of structure factors, atomic positional and thermal parameters for all atoms and other information in the printed form of the Standard Crystallographic File Structure of Brown (1983) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39992 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Fractional coordinates and, for non-hydrogen atoms, equivalent isotropic temperature factors,* U_{eq} ($Å^2 \times 10^3$), *with e.s.d.'s in parentheses*

U_{eq} is the average of the eigenvalues of **U**.

	x	y	z	U_{eq}
Compound (I)				
C(1)	0·2113 (21)	0·5964 (7)	0·0823 (5)	57 (5)
C(2)	0·1942 (24)	0·6692 (7)	0·0432 (8)	82 (6)
C(3)	0·2908 (24)	0·6505 (8)	−0·0177 (7)	78 (6)
C(4)	0·1936 (24)	0·5780 (8)	−0·0485 (6)	65 (6)
C(5)	0·1985 (19)	0·5097 (7)	−0·0056 (5)	46 (5)
C(6)	0·1277 (19)	0·4310 (8)	−0·0295 (5)	52 (5)
C(7)	0·2004 (20)	0·3633 (7)	0·0080 (5)	46 (5)
C(8)	0·1130 (21)	0·3701 (8)	0·0713 (5)	54 (6)
C(9)	0·1712 (18)	0·4526 (7)	0·0963 (4)	40 (4)
C(10)	0·1079 (16)	0·5219 (7)	0·0564 (5)	45 (5)
C(11)	−0·0383 (21)	0·5982 (8)	−0·0708 (6)	71 (6)
C(12)	0·3348 (24)	0·5612 (8)	−0·1012 (6)	80 (6)
C(13)	−0·1260 (18)	0·3476 (7)	0·0768 (5)	64 (5)
O(14)	0·2480 (12)	0·3195 (4)	0·1088 (3)	56 (4)
C(15)	0·2595 (15)	0·2406 (7)	0·0997 (5)	46 (5)
O(16)	0·1569 (15)	0·2040 (5)	0·0654 (4)	93 (4)
C(17)	0·4325 (19)	0·2099 (7)	0·1404 (5)	59 (5)
C(18)	0·1062 (21)	0·4583 (7)	0·1615 (5)	63 (6)
C(19)	0·2837 (25)	0·4466 (7)	0·2052 (5)	63 (6)
O(20)	0·4335 (14)	0·5121 (5)	0·2023 (4)	58 (3)
C(21)	0·5880 (24)	0·5140 (10)	0·2428 (7)	65 (7)
O(22)	0·6056 (18)	0·4676 (6)	0·2815 (5)	101 (5)
C(23)	0·7258 (24)	0·5862 (8)	0·2373 (6)	52 (6)
C(24)	0·9141 (25)	0·5918 (8)	0·2729 (6)	58 (6)
C(25)	1·0463 (20)	0·6557 (10)	0·2673 (7)	61 (6)
C(26)	0·9956 (22)	0·7136 (8)	0·2311 (6)	63 (6)
C(27)	0·811 (3)	0·7123 (7)	0·1957 (5)	72 (6)
C(28)	0·6778 (22)	0·6461 (8)	0·1999 (5)	54 (5)
Br(29)	1·18035 (24)	0·80338 (9)	0·22391 (7)	86·5 (5)
C(30)	−0·1433 (18)	0·5343 (6)	0·0582 (5)	55 (5)
Compound (II)				
O(1)	0·6257 (5)	0·8556 (3)	0·93979 (23)	43·7 (13)
O(2)	1·2375 (6)	0·8223 (4)	0·9689 (3)	52·3 (13)
C(1)	1·0848 (9)	0·9793 (5)	0·7391 (4)	43·3 (20)
C(2)	1·1348 (9)	0·9870 (6)	0·6520 (4)	52·8 (22)
C(3)	1·0841 (10)	0·8788 (6)	0·6101 (4)	53·6 (22)
C(4)	0·8764 (10)	0·8470 (5)	0·6166 (4)	46·5 (20)
C(5)	0·8213 (8)	0·8490 (5)	0·7041 (3)	36·5 (18)
C(6)	0·6165 (9)	0·8182 (5)	0·7179 (3)	42·0 (19)
C(7)	0·5775 (8)	0·7983 (5)	0·8061 (4)	41·9 (18)
C(8)	0·6365 (8)	0·8932 (4)	0·8580 (3)	31·0 (15)
C(9)	0·8398 (8)	0·9277 (4)	0·8423 (3)	29·2 (16)
C(10)	0·8780 (8)	0·9536 (4)	0·7516 (3)	31·4 (16)
C(11)	0·8614 (13)	0·7262 (6)	0·5848 (4)	66 (3)
C(12)	0·7586 (11)	0·9240 (7)	0·5648 (4)	57·6 (23)
C(13)	0·5013 (8)	0·9944 (5)	0·8534 (4)	44·9 (17)
C(14)	0·7772 (8)	1·0630 (5)	0·7279 (3)	39·0 (17)
C(15)	0·9753 (8)	0·8393 (5)	0·8791 (3)	37·7 (17)
C(16)	1·0728 (9)	0·8847 (6)	0·9508 (4)	48·1 (21)
H(11)	1·159 (10)	0·919 (5)	0·759 (4)	
H(12)	1·129 (10)	1·063 (5)	0·770 (4)	
H(21)	1·261 (10)	1·005 (6)	0·645 (4)	
H(22)	1·066 (9)	1·048 (6)	0·627 (4)	
H(31)	1·163 (9)	0·812 (6)	0·639 (4)	
H(32)	1·121 (9)	0·895 (5)	0·552 (4)	
H(5)	0·894 (9)	0·783 (5)	0·736 (4)	
H(61)	0·581 (10)	0·737 (5)	0·687 (4)	
H(62)	0·541 (9)	0·882 (5)	0·702 (4)	
H(71)	0·659 (10)	0·729 (6)	0·823 (4)	
H(72)	0·455 (9)	0·776 (6)	0·811 (4)	
H(9)	0·874 (9)	1·008 (5)	0·870 (4)	
H(101)	0·757 (9)	0·853 (5)	0·951 (4)	
H(102)	1·220 (9)	0·761 (5)	0·970 (4)	
H(111)	0·888 (9)	0·719 (5)	0·531 (4)	
H(112)	0·725 (9)	0·700 (6)	0·582 (4)	
H(113)	0·946 (9)	0·673 (5)	0·619 (4)	
H(121)	0·614 (9)	0·903 (5)	0·567 (4)	
H(122)	0·794 (9)	0·905 (5)	0·508 (4)	
H(123)	0·791 (9)	1·006 (6)	0·577 (4)	
H(131)	0·372 (10)	0·980 (5)	0·881 (4)	
H(132)	0·477 (9)	1·012 (5)	0·800 (4)	
H(133)	0·544 (9)	1·069 (6)	0·893 (4)	
H(141)	0·655 (9)	1·056 (6)	0·712 (4)	
H(142)	0·848 (10)	1·095 (5)	0·683 (4)	
H(143)	0·775 (9)	1·112 (6)	0·767 (4)	
H(151)	1·087 (9)	0·820 (5)	0·835 (4)	
H(152)	0·898 (9)	0·762 (5)	0·896 (4)	
H(161)	0·986 (10)	0·889 (6)	1·000 (4)	
H(162)	1·147 (10)	0·970 (5)	0·946 (4)	

Table 3. *Bond distances and torsion angles*

(*a*) Interatomic distances (Å) with e.s.d.'s in parentheses

Compound (I)			
C(1)—C(2)	1·536 (18)	C(10)—C(30)	1·557 (15)
C(1)—C(10)	1·542 (16)	O(14)—C(15)	1·366 (14)
C(2)—C(3)	1·550 (23)	C(15)—O(16)	1·187 (14)
C(3)—C(4)	1·546 (20)	C(15)—C(17)	1·507 (15)
C(4)—C(5)	1·525 (18)	C(18)—C(19)	1·494 (19)
C(4)—C(11)	1·552 (19)	C(19)—O(20)	1·450 (16)
C(4)—C(12)	1·513 (19)	O(20)—C(21)	1·328 (18)
C(5)—C(6)	1·517 (18)	C(21)—O(22)	1·193 (20)
C(5)—C(10)	1·540 (17)	C(21)—C(23)	1·502 (22)
C(6)—C(7)	1·509 (17)	C(23)—C(24)	1·418 (21)
C(7)—C(8)	1·551 (17)	C(23)—C(28)	1·367 (19)
C(8)—C(9)	1·563 (17)	C(24)—C(25)	1·368 (21)
C(8)—C(13)	1·522 (17)	C(25)—C(26)	1·329 (21)
C(8)—O(14)	1·475 (15)	C(26)—C(27)	1·395 (21)
C(9)—C(10)	1·545 (16)	C(26)—Br(29)	1·915 (13)
C(9)—C(18)	1·548 (16)	C(27)—C(28)	1·399 (20)
Compound (II)			
O(1)—C(8)	1·468 (6)	C(5)—C(10)	1·544 (8)
O(2)—C(16)	1·439 (8)	C(6)—C(7)	1·549 (9)
C(1)—C(2)	1·533 (9)	C(7)—C(8)	1·500 (8)
C(1)—C(10)	1·542 (9)	C(8)—C(9)	1·551 (8)
C(2)—C(3)	1·521 (10)	C(8)—C(13)	1·555 (8)
C(3)—C(4)	1·553 (10)	C(9)—C(10)	1·602 (8)
C(4)—C(5)	1·545 (8)	C(9)—C(15)	1·570 (8)
C(4)—C(11)	1·545 (9)	C(10)—C(14)	1·549 (8)
C(4)—C(12)	1·533 (10)	C(15)—C(16)	1·513 (9)
C(5)—C(6)	1·546 (8)		

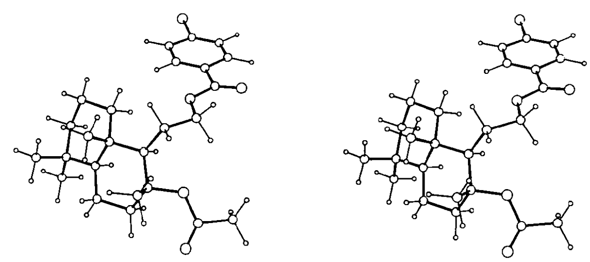
(*b*) Torsion angles (°) with e.s.d.'s in parentheses around rings *A* and *B*; angles start at the junction bond C(5)—C(10) and are given in counter-clockwise order

Compound (I)

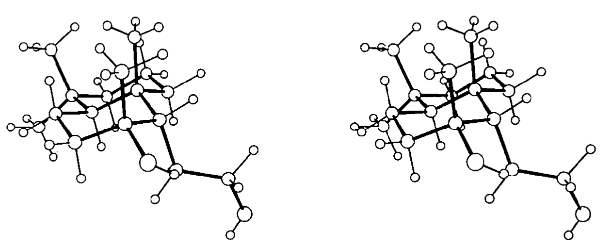
Ring *A*: −50 (1); 51 (1); −54 (1); 54 (2); −50 (1); 49 (1)
Ring *B*: 54 (1); −58 (1); 58 (1); −52 (1); 50 (1); −52 (1)

Compound (II)

Ring *A*: −50·1 (7); 52·6 (6); −57·4 (4); 58·0 (7); −52·1 (7); 48·8 (7)
Ring *B*: 57·9 (6); −57·2 (6); 53·4 (6); −51·4 (6); 53·1 (6); −56·1 (5)



(I)



(II)

Fig. 1. Stereopairs showing the configuration of the molecules.

six-membered rings adopt almost the same conformation as the (+)-8β analogue (Bernardinelli, Dunand, Flack, Yvon, Giersch & Ohloff, 1984). The main difference between the two structures occurs in the orientation of the *p*-bromobenzoyloxy fragment, due to the position of the acetoxy group. The values of the torsional angles C(9)—C(18)—C(19)—O(20) and C(18)—C(19)—O(20)—C(21) are respectively: 68·6 (1·3) and 173·3 (1·1)° for the α form and −174·5 (5) and 89·8 (7)° for the β form.

(II): Even with all Friedel pairs measured, the very small anomalous-dispersion contribution from the O atoms means that the absolute configuration of this molecule should be difficult to determine. This is borne out in practice by the absolute-structure parameter refining to $x = -0{\cdot}6$ (2·0), a value which does not significantly select one configuration from another. More precise measurements of sensitive reflections with a longer wavelength are called for. However, the absolute configuration of (II) can be deduced from its precursors (Lucius, 1960; Ruzicka, Seidel & Engel, 1942) and corresponds to that shown in Fig. 1. Hydrogen bonds occur between O(1)···O(2^{i}) 2·879 (6) and O(2)···O(1^{ii}) 2·754 (6) Å and lead to the presence of two short H···H intermolecular contacts: H(131)···H(162^{i}) 1·98 (10) and H(101)···H(102^{ii}) 1·93 (9) Å. [Symmetry code: (i) $x-1, y, z$; (ii) $\frac{1}{2}+x, \frac{3}{2}-y, 2-z$.]

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Structure of the Hydrogen Oxalate of Formamide Oxime, $CH_5N_2O^+.C_2HO_4^-$, at 105 K

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Abstract. $M_r = 150{\cdot}10$, monoclinic, Pc, $a = 3{\cdot}530$ (1), $b = 9{\cdot}642$ (3), $c = 16{\cdot}706$ (6) Å, $\beta = 91{\cdot}07$ (3)°, $V = 568{\cdot}5$ (4) Å^3 at 105 K, $Z = 4$, $D_x = 1{\cdot}753$ (1), D_m(293 K) = 1·701 (5) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}158$ mm^{-1}, $F(000) = 312$, final $R = 0{\cdot}028$ for 2259 unique observed reflections. The crystals are built up of hydrogen oxalate ions, $C_2HO_4^-$, and N^1-hydroxyformamidinium ions, H_2N^+=CH—NHOH, the protonated form of a tautomer of formamide oxime. The anions are connected by short asymmetric OH···O bonds into infinite chains, which are interlinked by the cations. The conformation of the cation H_2N^+=CH—NH—OH is synperiplanar. The H-bonding system includes two-, three-, and four-center bonds (*i.e.* 'linear', 'bifurcated', and 'trifurcated' H bonds).

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Introduction. Formamide oxime inhibits DNA synthesis in cells and bacteria by the same mechanism as hydroxyurea, *i.e.* by inhibition of the enzyme ribonucleotide reductase, but was found to be less potent in accordance with a lower one-electron oxidizability (Kjøller Larsen, Sjöberg & Thelander, 1982). The compound has been shown to possess antitumor activity against L1210 leukemia (Flora, van't Riet & Wampler, 1978), and, together with hydroxyurea, to induce virus expression, probably *via* damage to cellular DNA (Rascati & Tennant, 1978).

In solution formamide oxime has been proposed to exist as an equilibrium between two tautomers, H_2N—CH=N—OH ⇌ HN=CH—NH—OH, but crystallizes in the amide oxime form (*cf.* Jeffrey, Ruble, McMullan, DeFrees & Pople, 1981, and references therein).