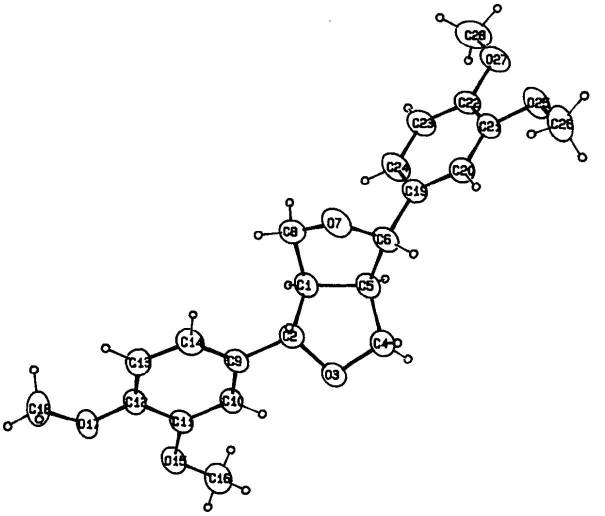

Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are represented by circles of arbitrary radius.

Related literature. Description of lignanes (Haworth, 1936); synthesis of (+), (−) and (±) forms of pinoresinol dimethyl ether (Erdtman, 1936); reaction with bromine to yield dibromo derivatives (Erdtman, 1935); characterization of lignanes having a 2,6-diaryl-*cis*-3,7-dioxabicyclo[3.3.0]octane structure (Adjangba, 1963; Hearon & MacGregor, 1955); unit-cell and space-group determination of dibromo and diiodo derivatives of pinoresinol dimethyl ether, establishing twofold molecular symmetry (Wang Lund, 1960); crystal-structure determination of the lignane (−)-syringaresinol (Bryan & Fallon, 1976), crystal-structure determination of the lignane (−)-3,6-bis(3,4-dimethoxyphenyl)tetrahydro-1*H*,3*H*-furo[3,4-*c*]furan-1,4-diol (Ghisalberti, Jefferies, Skelton & White, 1987).

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Structure of the Perchlorate Salt of Hetisine 13-*O*-Acetate

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Abstract. $C_{22}H_{30}NO_4^+.ClO_4^-$, $M_r = 471{\cdot}94$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}835\,(1)$, $b = 15{\cdot}736\,(1)$, $c = 11{\cdot}342\,(1)$ Å, $V = 2112{\cdot}4\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}48$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}24$ mm^{-1}, $F(000) = 1000$, $T = 294$ K, final $R = 0{\cdot}055$ for 1841 reflections. The perchlorate salt of this hitherto unknown 13-*O*-acetyl derivative of the alkaloid hetisine was examined to confirm the gross structure and relative configuration.

Experimental. Colourless plate, 0·15 × 0·30 × 0·50 mm, Enraf–Nonius CAD-4F diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice

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Table 1. *Positional* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10$) *for hetisine* 13-*O*-*acetate*.$HClO_4$

B_{eq} is defined as 1/3 the trace of the B_{ij} matrix.

	x	y	z	B_{eq}
C(1)	−303 (5)	3628 (3)	1749 (6)	28 (3)
C(2)	−99 (5)	3686 (4)	433 (6)	36 (3)
C(3)	−573 (5)	2939 (4)	−247 (6)	34 (3)
C(4)	−295 (5)	2061 (4)	278 (6)	29 (3)
C(5)	−558 (5)	2003 (3)	1602 (5)	27 (3)
C(6)	170 (5)	1265 (3)	2020 (5)	28 (3)
C(7)	235 (5)	1101 (3)	3330 (6)	30 (3)
C(8)	495 (5)	1920 (3)	3994 (5)	28 (3)
C(9)	−342 (5)	2635 (3)	3614 (6)	28 (3)
C(10)	−18 (5)	2757 (3)	2291 (6)	26 (3)
C(11)	−202 (5)	3345 (4)	4549 (6)	32 (3)
C(12)	965 (6)	3299 (4)	5131 (6)	35 (3)
C(13)	1901 (5)	3123 (3)	4239 (5)	31 (3)
C(14)	1602 (5)	2313 (3)	3508 (5)	26 (3)
C(15)	550 (6)	1752 (4)	5318 (6)	40 (3)
C(16)	957 (6)	2546 (4)	5964 (6)	36 (3)
C(17)	1310 (7)	2552 (4)	7061 (6)	49 (4)
C(18)	−930 (6)	1386 (4)	−429 (6)	41 (3)
C(19)	1003 (5)	1842 (4)	282 (5)	27 (3)
C(20)	1272 (5)	2510 (3)	2254 (5)	25 (3)
C(21)	2656 (5)	4498 (4)	3756 (7)	31 (3)
C(22)	2775 (6)	5124 (4)	2814 (6)	40 (3)
N	1254 (4)	1679 (3)	1562 (4)	27 (2)
O(2)	1091 (4)	3755 (2)	162 (4)	37 (2)
O(11)	−402 (4)	4207 (2)	4102 (4)	40 (2)
O(19)	2049 (3)	3815 (2)	3414 (3)	30 (2)
O(21)	3042 (4)	4560 (3)	4750 (4)	38 (2)
Cl	8463 (2)	4837 (1)	7319 (2)	47 (1)
O(5)	9420 (5)	5318 (4)	7551 (6)	100 (4)
O(6)	7992 (6)	4993 (6)	6210 (6)	120 (5)
O(7)	7681 (6)	4899 (7)	8239 (6)	128 (5)
O(8)	8745 (9)	3999 (4)	7299 (7)	142 (6)

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

C(1)—C(2)	1·515 (10)	C(1)—C(10)	1·540 (8)
C(2)—C(3)	1·513 (9)	C(2)—O(2)	1·446 (8)
C(3)—C(4)	1·539 (9)	C(4)—C(5)	1·537 (9)
C(4)—C(18)	1·529 (9)	C(4)—C(19)	1·574 (9)
C(5)—C(6)	1·522 (8)	C(5)—C(10)	1·558 (8)
C(6)—C(7)	1·511 (9)	C(6)—N	1·530 (8)
C(7)—C(8)	1·524 (8)	C(8)—C(9)	1·559 (8)
C(8)—C(14)	1·551 (9)	C(8)—C(15)	1·526 (9)
C(9)—C(10)	1·560 (9)	C(9)—C(11)	1·549 (9)
C(10)—C(20)	1·575 (8)	C(11)—C(12)	1·533 (10)
C(11)—O(11)	1·467 (7)	C(12)—C(13)	1·526 (9)
C(12)—C(16)	1·516 (9)	C(13)—C(14)	1·561 (8)
C(13)—O(19)	1·446 (7)	C(14)—C(20)	1·507 (9)
C(15)—O(16)	1·527 (9)	C(16)—C(17)	1·312 (9)
C(19)—N	1·504 (8)	C(20)—N	1·526 (7)
C(21)—C(22)	1·461 (9)	C(21)—O(19)	1·349 (7)
C(21)—O(21)	1·220 (9)		
Cl—O(5)	1·388 (6)	Cl—O(6)	1·398 (6)
Cl—O(7)	1·398 (7)	Cl—O(8)	1·360 (6)

C(2)—C(1)—C(10)	114·3 (5)	C(1)—C(2)—C(3)	113·3 (5)
C(1)—C(2)—O(2)	111·7 (5)	C(3)—C(2)—O(2)	108·1 (5)
C(2)—C(3)—C(4)	114·9 (6)	C(3)—C(4)—C(5)	112·8 (5)
C(3)—C(4)—C(18)	108·4 (5)	C(3)—C(4)—C(19)	114·0 (5)
C(5)—C(4)—C(18)	111·8 (5)	C(5)—C(4)—C(19)	100·5 (5)
C(18)—C(4)—C(19)	109·2 (5)	C(4)—C(5)—C(6)	103·6 (5)
C(4)—C(5)—C(10)	111·2 (5)	C(6)—C(5)—C(10)	101·1 (5)
C(5)—C(6)—C(7)	117·7 (5)	C(5)—C(6)—N	92·6 (4)
C(7)—C(6)—N	111·3 (5)	C(6)—C(7)—C(8)	110·6 (5)
C(7)—C(8)—C(9)	110·2 (5)	C(7)—C(8)—C(14)	109·4 (5)
C(7)—C(8)—C(15)	110·4 (5)	C(9)—C(8)—C(14)	98·7 (4)
C(9)—C(8)—C(15)	115·1 (5)	C(14)—C(8)—C(15)	112·5 (5)
C(8)—C(9)—C(10)	101·4 (4)	C(8)—C(9)—C(11)	105·3 (5)
C(10)—C(9)—C(11)	122·9 (5)	C(1)—C(10)—C(5)	112·8 (5)
C(1)—C(10)—C(9)	116·1 (5)	C(1)—C(10)—C(20)	114·9 (5)
C(5)—C(10)—C(9)	106·7 (4)	C(5)—C(10)—C(20)	101·3 (4)
C(9)—C(10)—C(20)	103·5 (5)	C(9)—C(11)—C(12)	111·0 (5)
C(9)—C(11)—O(11)	114·4 (5)	C(12)—C(11)—O(11)	109·7 (5)
C(11)—C(12)—C(13)	112·1 (5)	C(11)—C(12)—C(16)	107·5 (5)
C(13)—C(12)—C(16)	106·0 (5)	C(12)—C(13)—C(14)	109·7 (5)
C(12)—C(13)—O(19)	112·3 (5)	C(14)—C(13)—O(19)	107·4 (4)
C(8)—C(14)—C(13)	109·2 (5)	C(8)—C(14)—C(20)	101·4 (5)
C(13)—C(14)—C(20)	113·1 (4)	C(8)—C(15)—C(16)	110·1 (5)
C(12)—C(16)—C(15)	110·0 (6)	C(12)—C(16)—C(17)	125·7 (6)
C(15)—C(16)—C(17)	124·1 (6)	C(4)—C(19)—N	103·5 (5)
C(10)—C(20)—C(14)	106·0 (5)	C(10)—C(20)—N	102·3 (4)
C(14)—C(20)—N	108·2 (4)	C(22)—C(21)—O(19)	112·3 (6)
C(22)—C(21)—O(21)	125·8 (6)	O(19)—C(21)—O(21)	122·0 (6)
C(6)—N—C(19)	103·5 (4)	C(6)—N—C(20)	101·6 (4)
C(19)—N—C(20)	110·7 (4)	C(13)—O(19)—C(21)	118·6 (5)
O(5)—Cl—O(6)	113·6 (4)	O(5)—Cl—O(7)	111·1 (5)
O(5)—Cl—O(8)	109·3 (5)	O(6)—Cl—O(7)	113·3 (4)
O(6)—Cl—O(8)	104·7 (5)	O(7)—Cl—O(8)	104·1 (5)

parameters from least-squares refinement of 25 reflections with $12{\cdot}1 \le \theta \le 20{\cdot}0°$, space group determined from systematic absences and successful solution and refinement of the structure; 2115 unique reflections collected; 1435 considered observed at the $3\sigma(I)$ level [$\sigma(I)$ from counting statistics]; $\theta_{\max} = 25°$, $\omega/2\theta$ scans, scan range $1{\cdot}5(0{\cdot}76 + 0{\cdot}347\tan\theta)°$, scan speed 3·4 ranging to 0·74° min^{-1}; three standard reflections ($\bar{2}6\bar{3}$, $\bar{6}10$, $0\bar{7}\bar{3}$) measured every 2400 s of X-ray exposure time, max. variation 0·5%; data collected: $+h+k+l$ to max. indices of 14,18,13; data corrected for background and Lp, no correction for absorption. Structure solved by direct methods, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), and difference Fourier maps, refined by full-matrix least squares based on F, minimizing the function $\sum w(|F_o| - |F_c|)^2$; w defined as $[\sigma^2(F_o) + 0{\cdot}0001(F_o^2)]^{-1}$; *XRAY*76 system of programs (Stewart, 1976); H atoms included in positions determined by difference Fourier map with isotropic thermal parameters set to 1·1 times B_{eq} of the bonded atom; H-atom parameters not refined; all non-H refined with anisotropic thermal parameters. Convergence achieved in final cycles by alternately refining anion and cation; model converged with 1841 reflections [observed plus 406 for which $I_c > 3\sigma(I_o)$; 274 have $I_c < 3\sigma(I_o)$], 46 (anion) and 243 (cation) variables, $R = 0{\cdot}055$, $wR = 0{\cdot}056$, max. $(\Delta/\sigma) = 0{\cdot}01$, $S = 0{\cdot}91$, isotropic extinction parameter could not be refined, max. residual electron density 0·36 e Å^{-3} associated with Cl. Scattering factors were those of Cromer & Mann (1968) for non-H atoms, and Stewart, Davidson & Simpson (1965) for H atoms. Anomalous-dispersion corrections included for non-H atoms (*International Tables for X-ray Crystallography*, (1974), refinement of both enantiomorphs showed no significant difference in R values, chirality of parent alkaloid assumed. Atomic parameters may be found in Table 1 and bond lengths and

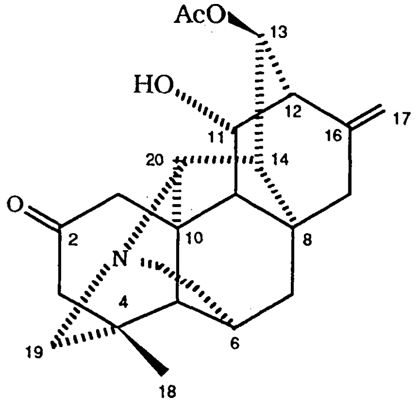

Fig. 1. Chemical numbering scheme.

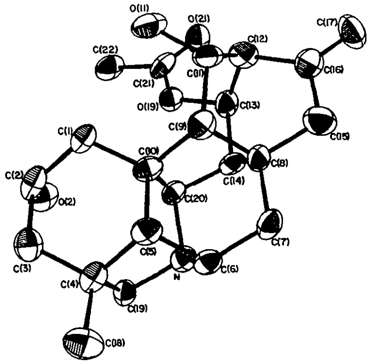

Fig. 2. *ORTEP* plot (Johnson, 1965) of hetisine 13-*O*-acetate. Thermal ellipsoids are drawn at the 50% probability level.

angles are given in Table 2.* Fig. 1 shows the chemical numbering scheme and an *ORTEP* plot is presented in Fig. 2. Relative stereochemistry and skeletal framework determined in this study confirm preliminary identification of the new diterpenoid alkaloid as the 13-*O*-acetyl derivative of hetisine.

Related literature. A preliminary report of this work and the isolation and spectroscopic identification of the title compound have been published (Benn, Richardson & Majek, 1986). The absolute configuration of the structure reported herein was not determined but the stereochemistry presented is in agreement with that determined for the parent alkaloid, hetisine (Przybylska, 1962).

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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 52307 (12 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 3-Oxa-1,5-pentanediyl Bis(*p*-toluenesulfonate)

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Abstract. $C_{18}H_{22}O_7S_2$, M_r = 414·5, monoclinic, $C2/c$, a = 23·772 (8), b = 5·472 (5), c = 15·284 (6) Å, β = 89·61 (3)°, V = 1987·9 Å^3, Z = 4, D_x = 1·385 g cm^{-3}, Mo $K\alpha$ radiation, λ = 0·7107 Å, μ = 29·06 cm^{-1}, $F(000)$ = 872, T = 294 K, R = 0·045 for 538 unique observed reflections [$I > 3\sigma(I)$]. This linear polyether

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