suivant une géométrie standard. Facteur de température B global affiné pour les H = 4,7 (2) Å^2, excepté les H du méthyl où B global = 7,6 (5) Å^2. Facteur R final = 0,035 pour 1740 réflexions observées [$I > 2{,}5\sigma(I)$]. $wR = 0{,}041$ où $w = 1/[\sigma^2(F) + 0{,}0034F^2]$. Δ/σ des paramètres finals ≤ 0,05. Limites des $\Delta\rho$ dans la carte de Fourier-différence finale: −0,30 et 0,13 e Å^{-3}. Les paramètres atomiques sont donnés dans le Tableau 1.* La liste des distances et des angles des liaisons se trouve dans le Tableau 2. La Fig. 1 montre une vue en perspective de la molécule avec la numérotation atomique. La Fig. 2 représente la structure cristalline.

Littérature associée. La structure du composé (I) a été réalisée dans le cadre de notre étude sur les récepteurs à dopamine. Voir par exemple la structure de la clothiapine (Sbit, Dupont, Dideberg, Liégeois & Delarge, 1987). Des analogues hétérocycliques originaux sont en voie d'expérimentation.

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* Les listes des facteurs de structure calculés et observés, les facteurs d'agitation thermique anisotrope, les coordonnées des atomes H et des paramètres des plans moyens ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 55076: 10 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre. [Référence de CIF: PA0271]

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Structure of Xindongnin B

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Abstract. *ent*-3β,7β,11β-Trihydroxy-15-oxokaur-16-en-6α-yl acetate, $C_{22}H_{32}O_6$, M_r = 392.50, orthorhombic, $P2_12_12_1$, a = 6.739 (5), b = 16.963 (6), c = 17.094 (6) Å, V = 1954.0 (3) Å^3, Z = 4, D_x = 1.334 g cm^{-3}, λ(Cu $K\alpha$) = 1.5410 Å, μ = 7.431 cm^{-1}, $F(000)$ = 848, room temperature, final R = 0.039 for 1055 observed reflections. The structure is composed of discrete molecules held together by hydrogen bonding. Each molecule contains three cyclohexane rings (A, B, C) in chair conformations, with the rings connected *trans* to each other. One five-membered ring is bridged by two C atoms to form an α-methylenecyclopentanone ring (D) with two unsaturated side groups. The five-membered ring has an envelope configuration with a C atom at the flap. This arrangement plays a key role in the antineoplastic activity of the compound.

Experimental. Single crystals suitable for X-ray work were crystallized from ethanol solution. A transparent crystal (0.2 × 0.3 × 0.3 mm) was used for X-ray measurements on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions were obtained from least-squares refinement of 25 reflections in the interval $15.7 < \theta < 29.7°$. The intensities of 1944 reflections

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3)]$.

	x	y	z	B_{eq}(Å^2)
O(1)	0.583 (1)	0.8500 (3)	0.2796 (4)	4.7 (2)
O(2)	0.099 (1)	0.9723 (3)	0.4959 (3)	3.4 (1)
O(3)	0.570 (1)	1.0615 (3)	0.4573 (3)	3.8 (1)
O(4)	0.274 (1)	1.1885 (3)	0.2366 (3)	3.8 (1)
O(5)	0.519 (1)	1.2117 (3)	0.4117 (4)	4.2 (2)
O(6)	0.188 (1)	0.8955 (4)	0.5962 (3)	4.9 (2)
C(1)	0.263 (1)	0.9595 (4)	0.2411 (4)	3.0 (2)
C(2)	0.252 (2)	0.8693 (5)	0.2335 (5)	3.4 (2)
C(3)	0.381 (2)	0.8292 (4)	0.2941 (5)	3.7 (2)
C(4)	0.322 (2)	0.8514 (4)	0.3770 (5)	3.3 (2)
C(5)	0.318 (1)	0.9437 (4)	0.3832 (4)	2.5 (2)
C(6)	0.300 (1)	0.9760 (5)	0.4654 (4)	2.8 (2)
C(7)	0.362 (1)	1.0621 (5)	0.4695 (5)	3.2 (2)
C(8)	0.252 (1)	1.1141 (4)	0.4108 (4)	2.3 (2)
C(9)	0.250 (1)	1.0775 (4)	0.3274 (4)	2.4 (2)
C(10)	0.192 (1)	0.9885 (4)	0.3213 (4)	2.5 (2)
C(11)	0.137 (1)	1.1309 (4)	0.2692 (5)	3.0 (2)
C(12)	−0.039 (2)	1.1771 (4)	0.3034 (5)	3.2 (2)
C(13)	0.001 (1)	1.2078 (4)	0.3848 (5)	2.9 (2)
C(14)	0.043 (2)	1.1377 (4)	0.4383 (4)	2.8 (2)
C(15)	0.344 (1)	1.1968 (4)	0.4043 (4)	2.8 (2)
C(16)	0.185 (2)	1.2549 (4)	0.3903 (5)	2.9 (2)
C(17)	0.217 (2)	1.3315 (5)	0.3877 (5)	3.6 (2)
C(18)	0.126 (2)	0.8100 (5)	0.3978 (5)	4.2 (2)
C(19)	0.477 (2)	0.8186 (5)	0.4326 (5)	4.7 (3)
C(20)	−0.035 (2)	0.9762 (4)	0.3269 (5)	3.5 (2)
C(21)	0.062 (2)	0.9321 (5)	0.5612 (5)	3.5 (2)
C(22)	−0.148 (2)	0.9375 (6)	0.5848 (5)	5.3 (3)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and hydrogen-bond geometry* (Å,°)

O(1)—C(3)	1.431 (15)	C(6)—C(7)	1.520 (12)
O(2)—C(6)	1.453 (9)	C(7)—C(8)	1.529 (11)
O(2)—C(21)	1.331 (10)	C(8)—C(9)	1.555 (10)
O(3)—C(7)	1.418 (10)	C(8)—C(14)	1.538 (14)
O(4)—C(11)	1.454 (9)	C(8)—C(15)	1.538 (10)
O(5)—C(15)	1.216 (9)	C(9)—C(10)	1.564 (10)
O(6)—C(21)	1.210 (13)	C(9)—C(11)	1.545 (10)
C(1)—C(2)	1.537 (11)	C(10)—C(20)	1.541 (15)
C(1)—C(10)	1.534 (10)	C(11)—C(12)	1.536 (13)
C(2)—C(3)	1.512 (15)	C(12)—C(13)	1.508 (12)
C(3)—C(4)	1.519 (13)	C(13)—C(14)	1.526 (10)
C(4)—C(5)	1.570 (10)	C(13)—C(16)	1.481 (14)
C(4)—C(18)	1.542 (17)	C(15)—C(16)	1.472 (13)
C(4)—C(19)	1.518 (16)	C(16)—C(17)	1.318 (11)
C(5)—C(6)	1.513 (10)	C(21)—C(22)	1.479 (19)
C(5)—C(10)	1.554 (10)		

C(6)—O(2)—C(21)	119.6 (7)	C(8)—C(9)—C(10)	116.6 (6)
C(2)—C(1)—C(10)	112.4 (6)	C(8)—C(9)—C(11)	111.1 (6)
C(1)—C(2)—C(3)	111.3 (7)	C(10)—C(9)—C(11)	113.5 (6)
O(1)—C(3)—C(2)	108.5 (8)	C(1)—C(10)—C(5)	106.3 (5)
O(1)—C(3)—C(4)	110.4 (8)	C(1)—C(10)—C(9)	106.9 (5)
C(2)—C(3)—C(4)	112.3 (7)	C(1)—C(10)—C(20)	108.7 (6)
C(3)—C(4)—C(5)	108.4 (6)	C(5)—C(10)—C(9)	106.8 (5)
C(3)—C(4)—C(18)	109.0 (7)	C(5)—C(10)—C(20)	115.6 (6)
C(3)—C(4)—C(19)	108.3 (8)	C(9)—C(10)—C(20)	112.1 (6)
C(5)—C(4)—C(18)	114.9 (7)	O(4)—C(11)—C(9)	109.1 (6)
C(5)—C(4)—C(19)	109.7 (6)	O(4)—C(11)—C(12)	107.0 (5)
C(18)—C(4)—C(19)	106.3 (7)	C(9)—C(11)—C(12)	115.8 (7)
C(4)—C(5)—C(6)	115.1 (6)	C(11)—C(12)—C(13)	113.0 (8)
C(4)—C(5)—C(10)	116.9 (6)	C(12)—C(13)—C(14)	108.5 (6)
C(6)—C(5)—C(10)	114.4 (6)	C(12)—C(13)—C(16)	113.2 (7)
O(2)—C(6)—C(5)	113.0 (5)	C(14)—C(13)—C(16)	103.0 (7)
O(2)—C(6)—C(7)	106.3 (5)	C(8)—C(14)—C(13)	101.0 (7)
C(5)—C(6)—C(7)	111.6 (6)	O(5)—C(15)—C(8)	125.0 (6)
O(3)—C(7)—C(6)	105.0 (6)	O(5)—C(15)—C(16)	125.7 (6)
O(3)—C(7)—C(8)	112.8 (6)	C(8)—C(15)—C(16)	109.3 (7)
C(6)—C(7)—C(8)	113.0 (6)	C(13)—C(16)—C(15)	105.0 (6)
C(7)—C(8)—C(9)	112.1 (6)	C(13)—C(16)—C(17)	131.8 (11)
C(7)—C(8)—C(14)	113.2 (6)	C(15)—C(16)—C(17)	123.2 (11)
C(7)—C(8)—C(15)	112.3 (6)	O(2)—C(21)—O(6)	123.4 (9)
C(9)—C(8)—C(14)	112.2 (6)	O(2)—C(21)—C(22)	112.0 (8)
C(9)—C(8)—C(15)	107.5 (5)	O(6)—C(21)—C(22)	124.7 (8)
C(14)—C(8)—C(15)	98.7 (5)		

Hydrogen bonds

	O···O	O—H···O
O(3)—H(3)···O(5)	2.69	149.5
O(1)—H(1)···O(4^{i})	2.92	162.4
O(4)—H(4)···O(6^{ii})	2.80	164.4

Symmetry code: (i) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, 2 - y, -\frac{1}{2} + z$.

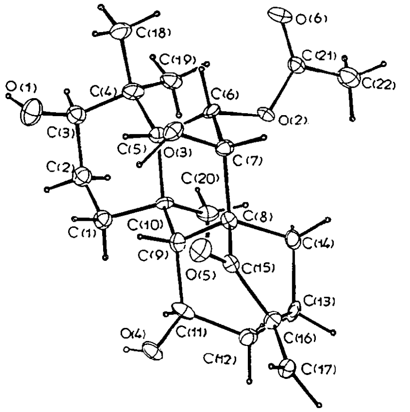

Fig. 1. Perspective drawing of the molecule with atom numbering.

with $1 \leq \theta \leq 65°$ (h 0→7, k 0→19, l 0→20) were measured in $\omega/2\theta$ scan mode with a maximum scan time of 60 s. Three standard reflections monitored every 2 h showed no significant variations. The data were corrected for Lp effects, and an absorption correction with transmission coefficients in the range 0.87–0.95 was made. 1055 reflections with $I > 3\sigma(I)$ were used for structure refinement. The structure was solved by direct methods and Fourier methods. All H atoms were located in $\Delta\rho$ maps at $R = 0.053$. The structure was refined on F by full-matrix least-squares calculations with anisotropic displacement parameters for non-H atoms and isotropic parameters for H atoms; the final discrepancy factors were $R = 0.039$ and $wR = 0.041$ with unit weights; $S = 1.076$ for 382 variables. Maximum shift/e.s.d. in final cycle was 1.57 and no residual electron density outside ±0.157 e Å^{-3} was found. Calculations were carried out on a PDP 11/34 computer using *SDP* (B. A. Frenz & Associates, Inc., 1982). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The fractional coordinates and equivalent isotropic thermal parameters for non-H atoms are given in Table 1, and selected bond lengths, bond angles and hydrogen-bond geometry are presented in Table 2.* Fig. 1 shows a perspective drawing of the molecule.

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* Lists of structure-factor amplitudes, anisotropic displacement parameters for non-H atoms, H-atom parameters, C—H bond lengths, least-squares-planes calculations and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54922 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0231]

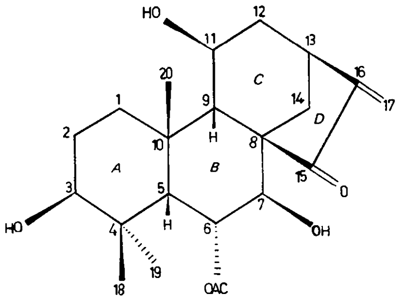

Fig. 2. Drawing of the structural formula.

Related literature. The title compound is one of the main components extracted from *Robodosia rubescens* (Hemsl.) Hara. Pharmacological tests show that xindongnin B has an antineoplastic action against Ehrlich ascites carcinoma cells, mouse sarcoma 180 and mouse lymphoma cell leukemia P388 for animals. The X-ray analysis indicates the structural formula given in Fig. 2. The molecular structure derived from elemental analysis and mass spectrometry (Sun, Ling, Fu, Zheng & Gao, 1985) gave an incorrect arrangement of functional groups. The skeleton is very similar to those found in rabdoserrin A and D (Wu, Chen, Jin & Cheng, 1986; Wu & Chen 1985), macrocalin A (Chen, Yong, Xu & Cheng, 1985), rabdophyllin G (Chen, Wu & Cheng, 1984), rubescensine A (Chen, Jin, Zhang & Li, 1983), and lungshengrabdosin (Luo, Luo, Sun & Lin, 1988). All these compounds have a cyclopentanone ring similar to that reported by Fujita, Takeda, Shingu, Kido & Taira (1982), in which the interior angles are smaller than the normal values expected for sp^2 or sp^3 hybrids, thereby leading to an increase of internal stress which may in turn cause the antineoplastic activity (Cheng, Lin & Xu, 1984).

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Absolute Configuration of (−)-α-Acetylmethadol Hydrochloride

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Abstract. β-[2-(Dimethylamino)propyl]-α-ethyl-β-phenylbenzeneethanol acetate ester hydrochloride, $C_{23}H_{32}NO_2^+.Cl^-.H_2O$, $M_r = 407.9$, monoclinic, $P2_1$, $a = 15.608\,(4)$, $b = 8.637\,(2)$, $c = 17.273\,(5)$ Å, $\beta = 97.71\,(2)°$, $V = 2307.4\,(1)$ Å^3, $Z = 4$ (two methadol hydrochlorides and two water molecules per asymmetric unit), $D_x = 1.17$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.54184$ Å, $\mu = 1.64$ mm^{-1}, $F(000) = 880$, $T = 295$ K, final $R = 0.069$, $wR = 0.064$ for 2559 independent observed reflections. Both asymmetric C atoms in the two independent methadol molecules are '*S*'. Of the two N—H moieties one acts as a hydrogen-bond donor to a Cl atom (N—H = 1.21, H···Cl = 1.85, N···Cl = 3.03 Å, N—H···Cl = 162.3°) and the other is a donor to a water molecule (N—H = 0.90, H···O = 2.06, N···O = 2.80 Å, N—H···O = 138.2°). In addition, both water molecules hydrogen bond to both Cl atoms with O···Cl distances in the range 3.06–3.36 Å. A comparison of torsion angles for the two independent methadol molecules indicates that there is very little stereochemical similarity between them.

Experimental. The original material was provided by the National Institute on Drug Abuse. Data were collected on a clear colorless plate (0.032 × 0.20 × 0.70 mm) grown by evaporation from nitromethane, using an automated Siemens *R*3*m*/*V* diffractometer with incident beam monochromator. 25 centered reflections within $31 \le 2\theta \le 81°$ were used for

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