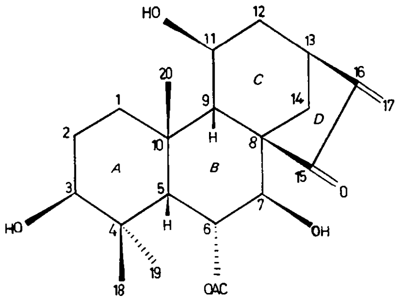

Fig. 2. Drawing of the structural formula.

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 *R3m/V* diffractometer with incident beam monochromator. 25 centered reflections within $31 \leq 2\theta \leq 81°$ were used for

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Table 1. *Atomic coordinates* (× 10⁴) *and equivalent isotropic displacement coefficients* (Å² × 10³)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cl(1)	5984 (2)	9202	5617 (2)	84 (1)
Cl(2)	1510 (2)	7455 (6)	5248 (2)	66 (1)
C(1)	4723 (5)	7399 (14)	8074 (5)	31 (3)
C(2)	4361 (5)	7518 (14)	7199 (5)	35 (3)
C(3)	4014 (5)	9088 (15)	6890 (5)	37 (3)
N(4)	4169 (5)	9277 (14)	6053 (4)	41 (3)
C(5)	3909 (8)	10825 (16)	5683 (7)	68 (5)
C(6)	3796 (8)	8017 (17)	5514 (6)	62 (5)
C(7)	3039 (5)	9388 (19)	6929 (6)	57 (4)
C(8)	4875 (5)	5604 (13)	8216 (5)	28 (3)
C(9)	5265 (7)	5101 (14)	9023 (6)	46 (4)
C(10)	5280 (7)	3318 (13)	9103 (7)	51 (4)
O(11)	5461 (4)	5120 (11)	7684 (3)	39 (2)
C(12)	5228 (7)	3918 (17)	7212 (6)	51 (5)
O(13)	4572 (5)	3167 (13)	7223 (5)	86 (4)
C(14)	5913 (8)	3562 (21)	6700 (8)	100 (7)
C(15)	5599 (5)	8228 (13)	8306 (5)	31 (3)
C(16)	6126 (5)	8621 (14)	7757 (6)	45 (4)
C(17)	6945 (6)	9333 (17)	7977 (7)	61 (5)
C(18)	7212 (6)	9592 (16)	8751 (7)	61 (5)
C(19)	6697 (6)	9227 (16)	9304 (6)	54 (4)
C(20)	5892 (6)	8536 (13)	9077 (6)	36 (4)
C(21)	4045 (5)	7977 (14)	8557 (5)	30 (3)
C(22)	3360 (5)	7057 (15)	8755 (5)	42 (4)
C(23)	2730 (6)	7675 (18)	9142 (6)	55 (4)
C(24)	2729 (6)	9201 (18)	9359 (6)	52 (4)
C(25)	3397 (6)	10123 (16)	9185 (6)	48 (4)
C(26)	4024 (5)	9586 (15)	8789 (5)	38 (4)
C(51)	−56 (5)	6029 (14)	7463 (5)	29 (3)
C(52)	−352 (5)	6079 (14)	6579 (5)	36 (4)
C(53)	−1228 (5)	6924 (14)	6298 (6)	38 (4)
N(54)	−1190 (5)	7716 (14)	5516 (5)	48 (3)
C(55)	−1001 (7)	6767 (19)	4847 (6)	76 (6)
C(56)	−557 (6)	9076 (17)	5609 (8)	87 (6)
C(57)	−2032 (6)	5899 (18)	6188 (7)	71 (5)
C(58)	142 (5)	7702 (14)	7790 (5)	32 (3)
C(59)	894 (6)	8488 (14)	7461 (6)	47 (4)
C(60)	907 (9)	10251 (16)	7620 (10)	91 (7)
O(61)	369 (3)	7578 (11)	8613 (4)	40 (2)
C(62)	37 (7)	8528 (17)	9098 (8)	61 (5)
O(63)	−484 (5)	9523 (13)	8880 (5)	79 (4)
C(64)	348 (8)	8168 (19)	9937 (7)	79 (6)
C(65)	−786 (5)	5282 (14)	7838 (6)	33 (4)
C(66)	−904 (6)	3667 (15)	7763 (6)	44 (4)
C(67)	−1595 (6)	2932 (16)	8038 (6)	54 (5)
C(68)	−2174 (6)	3752 (17)	8400 (7)	58 (5)
C(69)	−2060 (7)	5366 (18)	8478 (7)	61 (5)
C(70)	−1385 (5)	6101 (15)	8200 (6)	41 (4)
C(71)	788 (5)	5059 (13)	7619 (6)	30 (3)
C(72)	1357 (7)	4877 (14)	7082 (5)	39 (4)
C(73)	2140 (5)	4099 (14)	7266 (6)	42 (4)
C(74)	2355 (6)	3496 (14)	8000 (7)	49 (4)
C(75)	1797 (6)	3635 (15)	8539 (6)	51 (4)
C(76)	1030 (5)	4441 (14)	8365 (5)	37 (3)
O(1*S*)	2550 (4)	4247 (13)	5398 (4)	63 (3)
O(2*S*)	7849 (6)	694 (16)	6314 (6)	126 (5)

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

C(1)—C(2)	1.544 (12)	C(1)—C(8)	1.582 (16)
C(1)—C(15)	1.548 (12)	C(1)—C(21)	1.518 (13)
C(2)—C(3)	1.530 (16)	C(3)—N(4)	1.506 (11)
C(3)—C(7)	1.555 (12)	N(4)—C(5)	1.514 (17)
N(4)—C(6)	1.498 (16)	C(8)—C(9)	1.509 (13)
C(8)—O(11)	1.442 (11)	C(9)—C(10)	1.546 (17)
O(11)—C(12)	1.340 (15)	C(12)—O(13)	1.214 (15)
C(12)—C(14)	1.508 (18)	C(15)—C(16)	1.379 (14)
C(15)—C(20)	1.374 (13)	C(16)—C(17)	1.425 (14)
C(17)—C(18)	1.363 (16)	C(18)—C(19)	1.366 (16)
C(19)—C(20)	1.398 (13)	C(21)—C(22)	1.409 (14)
C(21)—C(26)	1.448 (17)	C(22)—C(23)	1.369 (15)
C(23)—C(24)	1.371 (21)	C(24)—C(25)	1.377 (16)
C(25)—C(26)	1.348 (14)	C(51)—C(52)	1.535 (12)
C(51)—C(58)	1.567 (16)	C(51)—C(65)	1.527 (13)
C(51)—C(71)	1.554 (13)	C(52)—C(53)	1.570 (12)
C(53)—N(54)	1.522 (14)	C(53)—C(57)	1.526 (15)
N(54)—C(55)	1.479 (17)	N(54)—C(56)	1.529 (17)
C(58)—C(59)	1.530 (14)	C(58)—O(61)	1.421 (11)
C(59)—C(60)	1.547 (18)	O(61)—C(62)	1.327 (16)
C(62)—O(63)	1.207 (16)	C(62)—C(64)	1.498 (17)
C(65)—C(66)	1.410 (17)	C(65)—C(70)	1.386 (14)
C(66)—C(67)	1.389 (15)	C(67)—C(68)	1.363 (17)
C(68)—C(69)	1.409 (21)	C(69)—C(70)	1.372 (16)
C(71)—C(72)	1.376 (13)	C(71)—C(76)	1.398 (13)
C(72)—C(73)	1.393 (13)	C(73)—C(74)	1.370 (15)
C(74)—C(75)	1.364 (15)	C(75)—C(76)	1.383 (14)

C(2)—C(1)—C(8)	104.2 (8)	C(2)—C(1)—C(15)	114.7 (8)
C(8)—C(1)—C(15)	107.7 (7)	C(2)—C(1)—C(21)	109.0 (7)
C(8)—C(1)—C(21)	109.7 (8)	C(15)—C(1)—C(21)	111.2 (8)
C(1)—C(2)—C(3)	117.8 (9)	C(2)—C(3)—N(4)	109.7 (8)
C(2)—C(3)—C(7)	115.8 (9)	N(4)—C(3)—C(7)	107.9 (7)
C(3)—N(4)—C(5)	115.7 (9)	C(3)—N(4)—C(6)	114.8 (9)
C(5)—N(4)—C(6)	108.6 (8)	C(1)—C(8)—C(9)	117.3 (8)
C(1)—C(8)—O(11)	106.2 (8)	C(9)—C(8)—O(11)	107.1 (7)
C(8)—C(9)—C(10)	111.6 (9)	C(8)—O(11)—C(12)	118.0 (8)
O(11)—C(12)—O(13)	124.4 (10)	O(11)—C(12)—C(14)	111.1 (10)
O(13)—C(12)—C(14)	124.4 (12)	C(1)—C(15)—C(16)	121.5 (8)
C(1)—C(15)—C(20)	120.6 (8)	C(16)—C(15)—C(20)	117.9 (8)
C(15)—C(16)—C(17)	121.3 (9)	C(16)—C(17)—C(18)	118.6 (10)
C(17)—C(18)—C(19)	121.1 (10)	C(18)—C(19)—C(20)	119.6 (10)
C(15)—C(20)—C(19)	121.6 (9)	C(1)—C(21)—C(22)	123.7 (10)
C(1)—C(21)—C(26)	120.7 (9)	C(22)—C(21)—C(26)	115.4 (9)
C(21)—C(22)—C(23)	121.1 (12)	C(22)—C(23)—C(24)	122.4 (11)
C(23)—C(24)—C(25)	117.8 (10)	C(24)—C(25)—C(26)	122.3 (13)
C(21)—C(26)—C(25)	121.0 (10)	C(52)—C(51)—C(58)	110.6 (9)
C(52)—C(51)—C(65)	107.0 (7)	C(58)—C(51)—C(65)	111.0 (8)
C(52)—C(51)—C(71)	108.8 (8)	C(58)—C(51)—C(71)	108.2 (7)
C(65)—C(51)—C(71)	111.2 (9)	C(51)—C(52)—C(53)	117.1 (8)
C(52)—C(53)—N(54)	110.3 (7)	C(52)—C(53)—C(57)	115.9 (10)
N(54)—C(53)—C(57)	106.3 (8)	C(53)—N(54)—C(55)	118.7 (11)
C(53)—N(54)—C(56)	110.7 (8)	C(55)—N(54)—C(56)	108.3 (9)
C(51)—C(58)—C(59)	113.5 (8)	C(51)—C(58)—O(61)	107.6 (9)
C(59)—C(58)—O(61)	107.7 (7)	C(58)—C(59)—C(60)	111.3 (10)
C(58)—O(61)—C(62)	121.2 (9)	O(61)—C(62)—O(63)	123.2 (11)
O(61)—C(62)—C(64)	112.3 (11)	O(63)—C(62)—C(64)	124.5 (13)
C(51)—C(65)—C(66)	118.4 (9)	C(51)—C(65)—C(70)	124.2 (11)
C(66)—C(65)—C(70)	117.2 (9)	C(65)—C(66)—C(67)	121.3 (10)
C(66)—C(67)—C(68)	120.8 (13)	C(67)—C(68)—C(69)	118.3 (11)
C(68)—C(69)—C(70)	121.2 (11)	C(65)—C(70)—C(69)	121.2 (12)
C(51)—C(71)—C(72)	123.6 (9)	C(51)—C(71)—C(76)	118.9 (8)
C(72)—C(71)—C(76)	117.3 (8)	C(71)—C(72)—C(73)	121.8 (9)
C(72)—C(73)—C(74)	119.6 (9)	C(73)—C(74)—C(75)	119.9 (9)
C(74)—C(75)—C(76)	120.6 (10)	C(71)—C(76)—C(75)	120.8 (9)

determining lattice parameters. $(\sin\theta/\lambda)_{\max} = 0.54$ Å^{-1}, range of *hkl*: $-17 \le h \le 9$, $0 \le k \le 9$, $-18 \le l \le 18$. Standards $\bar{4}11$, 105 and $\bar{3}21$, monitored every 97 reflections, showed a linear decrease of approximately 12% over data collection: $\theta/2\theta$ scan mode, scan width $[2\theta(K\alpha_1) - 1.0]$ to $[2\theta(K\alpha_2) + 1.0]$°, constant ω-scan rate (10.2° min^{-1}). 3570 reflections were measured, of which 2974 were unique ($R_{\rm int} = 3.1\%$) and 2559 observed with $F_o > 3\sigma(F_o)$. Data were corrected for Lorentz and polarization effects but not for absorption. The direct methods structure solution, and the full-matrix least-squares refinement used programs in *SHELXTL* (Sheldrick, 1980). The function $\sum w(|F_o| - |F_c|)^2$ was minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.000225$. Secondary-extinction parameter $p = 0.0038$ (5) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. The absolute configuration was determined by the method of Rogers (1981), the refined value of the variable multiplying f'' being 0.9 (2). There were 542 parameters refined: atomic coordinates and anisotropic thermal parameters for all non-H atoms; H atoms bonded to N atoms were located in a difference map and coordinates refined, all other H atoms were included using a riding model [coordinate shifts of C applied to attached H atoms, C—H distance set to 0.96 Å, H angles idealized,

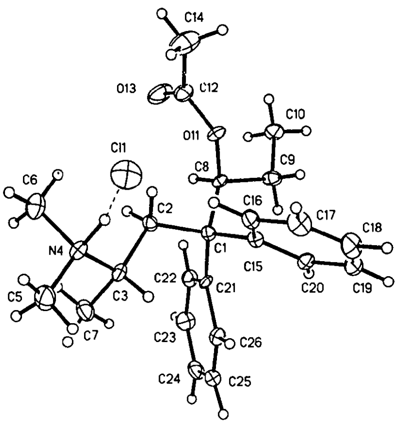

Fig. 1. A thermal ellipsoid plot of one of the two independent (−)-α-acetylmethadol molecules with ellipsoids drawn at the 20% probability level. The N—H···Cl hydrogen bond is also shown.

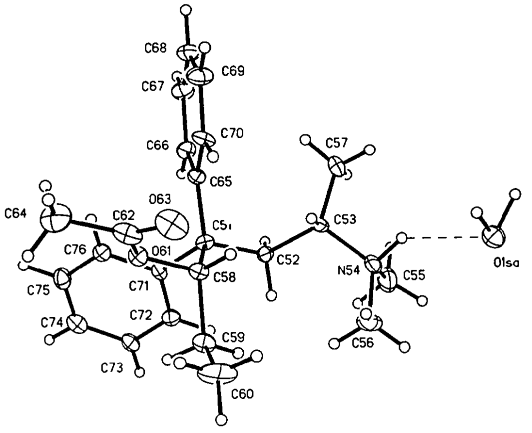

Fig. 2. A thermal ellipsoid plot of the other independent molecule of (−)-α-acetylmethadol hydrochloride with ellipsoids drawn at the 20% probability level. The N—H···O hydrogen bond is also shown.

fixed $U_{\mathrm{iso}}(\mathrm{H})$]. H atoms on the water molecules were not found. $(\Delta/\sigma)_{\max} = 0.35$, ratio of observations to parameters = 4.7:1, $R = 0.069$ ($R = 0.082$ for all data), $wR = 0.064$, $S = 1.49$. Final difference map excursions were 0.32 and −0.29 e Å^{-3}. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atom numbering for Tables 1, atom coordinates, and 2, bond distances and angles, follows that shown in Figs. 1 and 2.*

Related literature. The structures of methadone and several of its derivatives have been reported (Hanson & Ahmed, 1958; Bürgi, Dunitz & Shefter, 1973; Bye, 1974; Shefter, 1974; Singh & Moreland, 1989). The structure of a racemic mixture of the title compound has also been reported (Shefter, 1974).

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55113 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0604]

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Structure of 3,6-Diacetyl-8-benzoylantakyatriol

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Abstract. 3,6-Diacetyl-8-benzoylantakyatriol, (2) [4,10-dimethyl-7-(1-methylethyl)cyclodeca-4,10-diene-3,6,8-triol 3,8-diacetate 8-benzoate], was isolated during chemotaxonomical investigation of *Ferula* and closely related taxa. $C_{26}H_{34}O_6$, $M_r = 442.56$, orthorhombic, $P2_12_12_1$, $a = 7.766\ (6)$, $b = 11.809\ (4)$,

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