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Structure of Modifications (I) and (II) of 13-Ethyl-17β-hydroxy-18,19-dinor-17α-pregna-4,15-dien-20-yn-3-one (Gestodene)

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Abstract. $C_{21}H_{26}O_2$, $M_r = 310{\cdot}43$, orthorhombic, $P2_12_12_1$, $Z = 4$; modification (I): $a = 6{\cdot}620$ (3), $b = 12{\cdot}66$ (1), $c = 20{\cdot}70$ (1) Å, $V = 1735$ (2) Å^3, $D_x = 1{\cdot}188$ g cm^{-3}; modification (II): $a = 7{\cdot}6427$ (7), $b = 11{\cdot}345$ (2), $c = 20{\cdot}214$ (3) Å, $V = 1752{\cdot}7$ (4) Å^3, $D_x = 1{\cdot}176$ g cm^{-3}. (I) and (II): λ(Cu $K\alpha$) = 1·54184 Å, μ(Cu $K\alpha$) = 6 cm^{-1}, $F(000) = 672$, room temperature. Gestodene crystallized from ethanol shows two different modifications. $R = 0{\cdot}056$ for (I) and 0·043 for (II), for 1501 and 1821 unique reflections with $I \geq 2{\cdot}5\sigma(I)$ respectively. For (I) the ethyl group is in the *trans* conformation relative to the *C/D* ring junction, whereas for (II) the conformation is *gauche* with the ethyl group positioned above the *D* ring. Molecular mechanics gives a steric energy difference of 5 kJ mol^{-1} between the minimized *trans* and *gauche* structures in favour of the latter. The *A* ring shows a normal 1α,2β-half-chair conformation in both modifications. The head-to-tail hydrogen-bonded steroid molecules are similarly packed in both crystal structures.

Introduction. Gestodene is a new orally active progestogen enabling fertility control at a very low dose (Losert, Casals-Stenzel & Buse, 1985). The structure differs from levonorgestrel, which has been used for a long time in oral contraception, by the introduction of a Δ^{15} unsaturation. Gestodene shows a higher affinity for the progesterone receptor than levonorgestrel (Spona, Scheider, Bieglmayer, Schroeder & Pirker, 1979), but a similar affinity for the androgen receptor (Bergink & Kloosterboer, 1985). Cleve, Frost, Hoyer, Rosenberg & Seeger (1986) studied the structure of gestodene by spectroscopic methods. In order to enable a better understanding of the structure–activity relationships of the progestogens the 3D structure of gestodene has been determined by X-ray diffraction and its molecular conformation is compared with that of levonorgestrel (DeAngelis, Doyne & Grob, 1975).

Experimental. Crystals of gestodene were obtained by slow evaporation from ethanol. Weissenberg photographs indicated two different orthorhombic modifications [(I) and (II)] with slightly different cell dimensions. Crystal aggregates of both forms were also observed. The approximate dimensions of the measured crystals were 0·75 × 0·6 × 0·6 (I) and 1·0 × 0·4 × 0·3 (II) mm. Data were measured on an Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation; lattice parameters were refined by least-squares fitting of eight reflections, for (I): ±3 ±5 ±8 with $2\theta \simeq 66{\cdot}8°$ and for (II): ±2 ±3 ±5 with $2\theta \simeq 40{\cdot}2°$; ω–2θ scan mode, $\Delta\omega = (0{\cdot}5 + 0{\cdot}15\tan\theta)°$ for (I) and $\Delta\omega = (0{\cdot}7 + 0{\cdot}15\tan\theta)°$ for (II). 5517 ($\pm h$, k, $\pm l$ maximum range 8, 15, 15) and 1947 (h, k, l maximum range 9, 13, 24) reflections were measured up to $\theta = 70°$ and the intensities of equivalent and Friedel-related reflections were merged to 1529 and 1927 independent observations of which 1501 and 1821 were considered observed [$I \geq 2{\cdot}5\sigma(I)$] for modifications (I) and (II) respectively. For (I) four standard reflections were measured periodically and these showed less than 5% variation in intensity. For (II) three reflections were monitored and showed a decrease in intensity of only 2%. Data were corrected for this variation in scattering power but not for absorption.

For modification (I) initial phases were obtained from tangent refinement of phases calculated with the coordinates of the isomorphous crystal structure of levonorgestrel (DeAngelis, Doyne & Grob, 1975; Crabbé & Schlemper, 1983). The structure of (II) was solved by direct methods by default run with the *SHELXS*86 program (Sheldrick, 1986). For C and O atoms positional and anisotropic thermal parameters were refined on F by full-matrix least-squares minimization. H atoms were included on calculated positions and refined riding on their bonded atoms, except the hydroxyl and ethynyl group H atoms, which were located on a difference map and refined positionally. The thermal motion for H atoms was described by one overall isotropic thermal parameter. In total 215 parameters were varied using the *SHELX*76 program (Sheldrick, 1976). The refinement for (I) converged at $R = 0{\cdot}056$ and $wR = 0{\cdot}069$ [$w = 1/\sigma^2(F)$] with $\langle\Delta/\sigma\rangle = 0{\cdot}06$ (6) for non-H-atom parameters and $\langle\Delta/\sigma\rangle = 0{\cdot}1$ (1) for H-atom parameters. For (II) final $R = 0{\cdot}043$ and $wR = 0{\cdot}050$ [$w = 1/\sigma^2(F)$] with

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Table 1. *Positional and equivalent isotropic thermal parameters* (Å²) *for non-*H *atoms with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

	Gestodene (I)				Gestodene (II)			
	x	y	z	U_{eq}	x	y	z	U_{eq}
O(3)	0·9526 (4)	0·5823 (2)	0·0118 (1)	0·0757 (8)	0·5259 (3)	0·8935 (2)	0·0061 (1)	0·0567 (6)
O(17)	0·2009 (4)	0·5481 (2)	−0·4570 (1)	0·0670 (8)	1·2676 (3)	0·9711 (2)	−0·4550 (1)	0·0435 (6)
C(1)	0·7987 (6)	0·4756 (3)	−0·1392 (2)	0·067 (1)	0·6430 (3)	0·9997 (3)	−0·1536 (1)	0·0407 (8)
C(2)	0·8636 (6)	0·4570 (3)	−0·0700 (2)	0·077 (2)	0·5724 (4)	1·0243 (3)	−0·0848 (1)	0·0427 (6)
C(3)	0·8335 (5)	0·5563 (3)	−0·0307 (2)	0·061 (1)	0·6249 (4)	0·9273 (3)	−0·0380 (1)	0·0417 (6)
C(4)	0·6537 (5)	0·6183 (3)	−0·0455 (2)	0·066 (1)	0·8016 (4)	0·8819 (3)	−0·0456 (1)	0·0443 (6)
C(5)	0·5315 (4)	0·5969 (3)	−0·0947 (2)	0·058 (1)	0·9062 (3)	0·9139 (2)	−0·0955 (1)	0·0347 (6)
C(6)	0·3340 (5)	0·6543 (4)	−0·1036 (2)	0·082 (2)	1·0981 (3)	0·8838 (3)	−0·0953 (1)	0·0427 (8)
C(7)	0·2899 (5)	0·6883 (3)	−0·1721 (2)	0·070 (1)	1·1659 (3)	0·8385 (2)	−0·1618 (1)	0·0357 (6)
C(8)	0·3195 (4)	0·5982 (2)	−0·2205 (2)	0·055 (1)	1·1121 (3)	0·9192 (2)	−0·2187 (1)	0·0300 (6)
C(9)	0·5374 (4)	0·5553 (2)	−0·2138 (1)	0·0463 (8)	0·9101 (3)	0·9314 (2)	−0·2190 (1)	0·0317 (6)
C(10)	0·5788 (5)	0·5145 (2)	−0·1454 (2)	0·0543 (8)	0·8435 (3)	0·9854 (2)	−0·1539 (1)	0·0320 (6)
C(11)	0·5854 (4)	0·4725 (2)	−0·2657 (2)	0·0567 (8)	0·8401 (3)	0·9998 (2)	−0·2791 (1)	0·0393 (8)
C(12)	0·5432 (4)	0·5107 (2)	−0·3350 (2)	0·0520 (8)	0·9153 (3)	0·9628 (3)	−0·3465 (1)	0·0397 (8)
C(13)	0·3244 (4)	0·5472 (2)	−0·3406 (2)	0·0477 (8)	1·1160 (3)	0·9562 (2)	−0·3437 (1)	0·0317 (6)
C(14)	0·2894 (4)	0·6344 (2)	−0·2897 (2)	0·051 (1)	1·1641 (3)	0·8719 (2)	−0·2867 (1)	0·0320 (6)
C(15)	0·0957 (4)	0·6851 (3)	−0·3133 (2)	0·060 (1)	1·3474 (3)	0·8335 (2)	−0·3039 (1)	0·0373 (6)
C(16)	0·0833 (4)	0·6738 (2)	−0·3767 (2)	0·064 (1)	1·3721 (4)	0·8437 (2)	−0·3690 (1)	0·0403 (6)
C(17)	0·2619 (4)	0·6102 (2)	−0·4028 (2)	0·0537 (8)	1·2122 (3)	0·8949 (2)	−0·4033 (1)	0·0350 (6)
C(18)	0·1724 (5)	0·4537 (3)	−0·3318 (2)	0·067 (1)	1·1855 (3)	1·0839 (2)	−0·3359 (1)	0·0370 (6)
C(20)	0·4211 (4)	0·6853 (2)	−0·4244 (2)	0·0537 (8)	1·1049 (4)	0·7975 (3)	−0·4321 (1)	0·0420 (8)
C(21)	0·5403 (5)	0·7454 (3)	−0·4442 (2)	0·065 (1)	1·0208 (5)	0·7232 (3)	−0·4563 (1)	0·057 (1)
C(22)	0·2104 (6)	0·3540 (2)	−0·3693 (2)	0·075 (1)	1·3791 (4)	1·1067 (3)	−0·3254 (2)	0·052 (1)

$\langle \Delta/\sigma \rangle = 0{\cdot}005$ (3) for non-H-atom parameters and $\langle \Delta/\sigma \rangle = 0{\cdot}015$ (7) for H-atom parameters. The residual electron density in final difference Fourier maps was within $\pm 0{\cdot}3$ e Å^{-3}. For (I) the mean square amplitude of vibration for H atoms refined to 0·091 (2) Å² and for (II) to 0·056 (2) Å². Scattering factors were taken from *SHELX*76.

Discussion. The final atomic parameters are given in Table 1.* Fig. 1 shows the conformations of the steroid molecules for both (I) and (II). Bond distances and bond angles are given in Table 2 and correspond to those observed in related structures (Griffin, Duax & Weeks, 1984). The mean differences in corresponding bond distances and bond angles for (I) and (II) are 0·006 (5) Å and 1 (1)° respectively. The symmetry of the rings is illustrated by the use of asymmetry parameters (*e.g.* Griffin, Duax & Weeks, 1984). The Δ^4 *A* ring has a normal (Duax, Fronckowiak, Griffin & Rohrer, 1982) 1α,2β-half-chair conformation with ΔC_2[C(1)–C(2)] = 4·2 (5) and 1·5 (3)° for (I) and (II) respectively. The *B* and *C* rings have the usual chair conformation. The *D* ring has a 13β-envelope conformation imposed by the Δ^{15} unsaturation with ΔC_s[C(13)] = 3·6 (3)° for (I) and 3·5 (2)° for (II). For levonorgestrel ΔC_s[C(13)] = 7·8°. The overall conformation of modification (I) of gestodene is similar to that of levonorgestrel and a least-squares fit of all non-H atoms resulted in a mean deviation for the fitted atoms of 0·07 (1) Å. A scan through the Cambridge Crystallographic Database showed that in the crystalline state all steroids with a saturated *D* ring and a 13β-ethyl group have the ethyl group *trans* relative to the *C*/*D* ring junction and that only one steroid with a Δ^{14} unsaturation has the ethyl group in a *gauche* conformation with the ethyl group above the *D* ring [*rac*-3-methoxy-18-methylestra-1,3,5(10),8,14-pentaen-17-one (Chekhlov, Ionov, Dononov & Ananchenko, 1983)]. For (I) the conformation of the

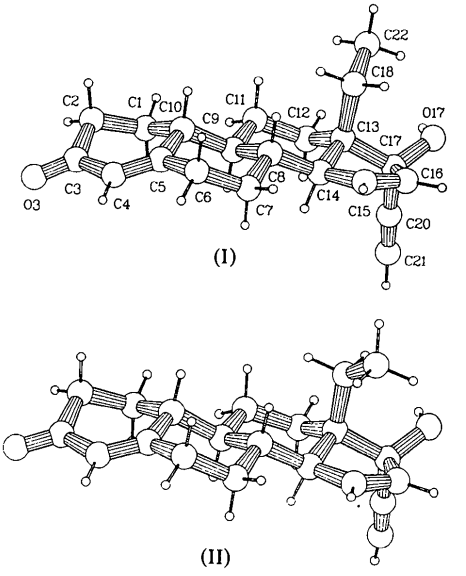

Fig. 1. Molecular conformation of gestodene, modifications (I) and (II).

*Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44261 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and bond angles* (°) *for non-*H *atoms with e.s.d.'s in parentheses*

	(I)	(II)		(I)	(II)
O(3)–C(3)	1·231 (3)	1·226 (4)	C(9)–C(10)	1·538 (3)	1·532 (5)
O(17)–C(17)	1·421 (3)	1·428 (4)	C(9)–C(11)	1·538 (3)	1·534 (4)
C(1)–C(2)	1·518 (3)	1·514 (6)	C(11)–C(12)	1·537 (3)	1·539 (6)
C(1)–C(10)	1·541 (3)	1·542 (5)	C(12)–C(13)	1·537 (3)	1·525 (4)
C(2)–C(3)	1·506 (4)	1·511 (6)	C(13)–C(14)	1·542 (3)	1·544 (5)
C(3)–C(4)	1·453 (4)	1·458 (5)	C(13)–C(17)	1·573 (3)	1·570 (5)
C(4)–C(5)	1·337 (3)	1·329 (5)	C(13)–C(18)	1·551 (3)	1·564 (4)
C(5)–C(6)	1·506 (3)	1·507 (5)	C(14)–C(15)	1·508 (3)	1·515 (4)
C(5)–C(10)	1·510 (3)	1·513 (5)	C(15)–C(16)	1·334 (3)	1·323 (6)
C(6)–C(7)	1·530 (3)	1·510 (6)	C(16)–C(17)	1·520 (4)	1·529 (4)
C(7)–C(8)	1·526 (3)	1·531 (5)	C(17)–C(20)	1·494 (4)	1·488 (4)
C(8)–C(9)	1·550 (3)	1·548 (4)	C(18)–C(22)	1·517 (4)	1·503 (5)
C(8)–C(14)	1·528 (3)	1·517 (6)	C(20)–C(21)	1·167 (5)	1·170 (5)

	(I)	(II)
C(2)–C(1)–C(10)	113·3 (3)	112·1 (2)
C(1)–C(2)–C(3)	110·0 (3)	110·3 (3)
O(3)–C(3)–C(4)	122·1 (4)	122·5 (3)
O(3)–C(3)–C(2)	121·7 (3)	121·3 (3)
C(2)–C(3)–C(4)	116·3 (3)	116·1 (2)
C(3)–C(4)–C(5)	123·2 (4)	122·6 (2)
C(4)–C(5)–C(10)	123·1 (3)	123·1 (2)
C(4)–C(5)–C(6)	121·6 (4)	121·2 (2)
C(6)–C(5)–C(10)	115·3 (3)	115·7 (2)
C(5)–C(6)–C(7)	114·8 (3)	113·8 (2)
C(6)–C(7)–C(8)	112·2 (3)	111·7 (2)
C(7)–C(8)–C(9)	108·8 (3)	109·0 (2)
C(7)–C(8)–C(14)	112·1 (2)	113·4 (2)
C(9)–C(8)–C(14)	108·2 (2)	106·7 (2)
C(8)–C(9)–C(10)	111·6 (2)	111·2 (2)
C(8)–C(9)–C(11)	111·7 (2)	113·2 (2)
C(10)–C(9)–C(11)	112·3 (2)	111·1 (2)
C(5)–C(10)–C(9)	111·8 (2)	110·5 (2)
C(1)–C(10)–C(5)	111·0 (3)	111·7 (2)
C(1)–C(10)–C(9)	110·7 (3)	112·0 (2)
C(9)–C(11)–C(12)	113·6 (2)	115·6 (2)
C(11)–C(12)–C(13)	109·8 (3)	110·7 (2)
C(12)–C(13)–C(17)	117·8 (3)	117·4 (2)
C(14)–C(13)–C(18)	111·4 (3)	114·9 (2)
C(17)–C(13)–C(18)	108·1 (3)	109·3 (2)
C(12)–C(13)–C(14)	107·9 (3)	107·2 (2)
C(12)–C(13)–C(18)	111·9 (2)	107·5 (2)
C(14)–C(13)–C(17)	99·0 (2)	100·8 (2)
C(8)–C(14)–C(15)	122·9 (3)	123·4 (2)
C(13)–C(14)–C(15)	102·1 (3)	103·2 (2)
C(8)–C(14)–C(13)	114·1 (2)	113·1 (2)
C(14)–C(15)–C(16)	109·1 (3)	109·5 (2)
C(15)–C(16)–C(17)	111·1 (3)	111·7 (2)
C(16)–C(17)–C(20)	108·5 (2)	109·7 (2)
O(17)–C(17)–C(16)	110·4 (2)	109·2 (2)
C(13)–C(17)–C(16)	100·5 (3)	101·3 (2)
O(17)–C(17)–C(13)	116·0 (2)	115·7 (2)
O(17)–C(17)–C(20)	108·4 (3)	109·1 (2)
C(13)–C(17)–C(20)	112·6 (3)	111·6 (2)
C(13)–C(18)–C(22)	117·9 (3)	120·5 (2)
C(17)–C(20)–C(21)	176·5 (4)	178·0 (3)

angular ethyl group is approximately *trans* with respect to the C(13)–C(14) bond. The torsion angle C(14)–C(13)–C(18)–C(22) [−168·9 (3)°] is close to the mean value of −165 (3)° observed for 13-ethyl steroids with a saturated *D* ring. However, for (II) this conformation is *gauche* with C(14)–C(13)–C(18)–C(22) = 55·2 (3)°. Force-field calculation with the *MMP*2 program (Allinger & Flanagan, 1983) showed that in case of Δ^{15} unsaturation the *gauche* conformation is more favourable by 5 kJ mol^{-1}, in contrast with a saturated *D* ring (*e.g.* levonorgestrel), where the *trans* conformation is the most stable one by 6 kJ mol^{-1} (see Fig. 2). It is doubtful whether this ethyl group in *gauche* conformation is of any significance for explaining the increased binding to the progesterone receptor. It is thought that the presence of π-electron density in ring *D* of gestodene might be responsible for the increased binding to the receptor.

It has been deduced from IR absorption studies of 17-ethynyl-17-hydroxy steroids dissolved in CCl_4 that the OH group is oriented in only one of the three possible rotameric states (Visser & van der Maas, 1983). Similar IR studies for gestodene and levonorgestrel confirmed this preferred orientation, which is identical to that found in their crystal structures.

Fig. 3 shows the molecular packing in a stereoview down **a**. The steroid molecules are hydrogen-bonded from head to tail forming infinite chains parallel to **b**. For (I): O(17)→O[3, ($\frac{3}{2}-x$, $1-y$, $-\frac{1}{2}+z$)] with O···O = 2·899 (4), O–H = 1·03 (5), H···O = 1·87 (5) Å and O–H···O = 175 (4)°; and for (II): O(17)→O[3, ($\frac{3}{2}-x$, $2-y$, $-\frac{1}{2}+z$)] with O···O = 2·830 (3), O–H = 0·85 (3), H···O = 1·98 (3) Å and O–H···O = 171 (3)°. In modification (I) there are several short intermolecular

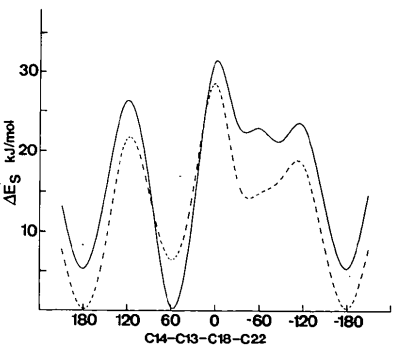

Fig. 2. Steric energy profiles for gestodene (full curve) and levonorgestrel (dashed) on rotation of the angular 13-ethyl group.

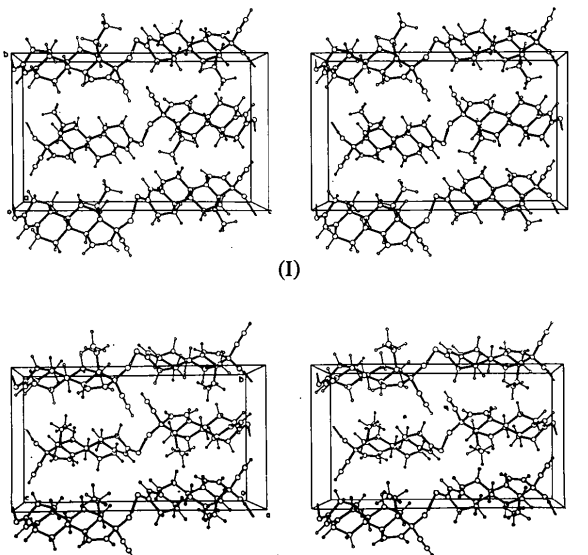

Fig. 3. Stereo packing diagrams of modifications (I) and (II).

H···H contact distances of ~2 Å involving H atoms of the ethyl group and H atoms bonded to C(7). In (II) no intermolecular H···H distances are present less than 2·1 Å.

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Structures of Strychnine Hydrogen (2*S*,3*S*)-Tartrate Trihydrate and Strychnine (2*R*,3*R*)-Tartrate Hexahydrate

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Abstract. (1): $C_{21}H_{23}N_2O_2^+.C_4H_5O_6^-.3H_2O$, $M_r=538{\cdot}49$, monoclinic, $P2_1$, $a=7{\cdot}374$ (2), $b=11{\cdot}713$ (2), $c=14{\cdot}293$ (2) Å, $\beta=97{\cdot}08$ (1)°, $V=1225{\cdot}1$ Å^3, $Z=2$, $D_x=1{\cdot}46$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu=1{\cdot}08$ cm^{-1}, $F(000)=572$, $T=293$ K, final $R=0{\cdot}0346$ for 1763 observed reflections [$I>2\sigma(I)$]. (2): $2C_{21}H_{23}N_2O_2^+.C_4H_4O_6^{2-}.6H_2O$, $M_r=926{\cdot}93$, triclinic, $P\bar{1}$, $a=7{\cdot}573$ (6), $b=7{\cdot}855$ (3), $c=19{\cdot}586$ (9) Å, $\alpha=87{\cdot}73$ (4), $\beta=81{\cdot}63$ (5), $\gamma=89{\cdot}59$ (5)°, $V=1151{\cdot}8$ Å^3, $Z=1$, $D_x=1{\cdot}34$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu=0{\cdot}95$ cm^{-1}, $F(000)=494$, $T=293$ K, final $R=0{\cdot}0786$ for 2509 observed reflections [$I>2\sigma(I)$]. (1) and (2) are similar structures both composed of alternate layers of alkaloid and counterion. The three independent strychnine ions in the structures are very similar, and the tartrate moieties have a similar conformation. The two strychnine packing types are different, that in (1) being similar to the packing seen in strychnine amino-acid salts, while the packing in (2) is most often seen in 'simple' strychnine mineral-acid salts.

Introduction. Strychnine is an indole alkaloid found in the seeds of *Strychnos nux vomica* and related plants. Aside from it use as a poison, it is important as a chiral in Pasteur resolution (Pasteur, 1853) of racemic acids. The two crystal structures described here are part of a large study on the diastereomeric salts of strychnine and the related alkaloid brucine. The tartrate salts provide examples of the changes in strychnine packing caused by small chiral counterions, and it was possible in this case to produce diastereomeric salts containing both enantiomers of tartaric acid. One of the products is a salt of the bivalent tartrate ion and the other of the univalent hydrogen tartrate (bitartrate) ion, a situation which is not uncommon in tartrate salt formation (Jacques, Collet & Wilen, 1981; Ladenburg, 1908).

Experimental. Approximately 0·05 g of a 2:1 mixture of strychnine and (2*S*,3*S*)-tartaric acid was dissolved in 0·5 ml of boiling water, and on cooling large needles of (1) were formed. An attempt to prepare (2) by the same method yielded crystals too small for X-ray diffraction, and the crystal eventually used for data collection was grown from a saturated 1:1 solution cooled over a period of eight hours. Both data sets were collected on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. Normal Lorentz–

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