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}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71069$ 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, $P1$, $a = 7{\cdot}573\,(6)$, $b = 7{\cdot}855\,(3)$, $c = 19{\cdot}586\,(9)$ 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}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71069$ 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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polarization corrections were applied, but no correction for absorption or extinction was made. Other experimental details are summarized in Table 1.

The structures were solved using the automated Patterson search methods of the *DIRDIF* system (Beurskens *et al.,* 1985). The orientation and translation searches used the entire non-hydrogen skeleton of a strychnine molecule as the search fragment. In (1), it is possible to locate all the non-H atoms of both the strychnine and the bitartrate moieties as well as two water molecules in the Fourier synthesis derived from the phases refined by *DIRDIF.* In (2), there are two independent strychnine cations in the asymmetric unit, and the non-H atoms of both of these were found in the *DIRDIF* Fourier synthesis. The tartrate ion was located in subsequent difference syntheses. The absolute configuration was established from the known strychnine configuration. Both structures were refined on *F* by

Table 1. *Experimental data*

	(1)	(2)
Crystal dimensions (mm)	0·25 × 0·12 × 0·1	0·4 × 0·3 × 0·2
Reflections to determine lattice parameters, θ range (°)	23, 3→12	25, 6→13
Max. $(\sin\theta)/\lambda$ (Å^{-1})	0·572	0·481
hkl range *h*:	−8, 8	0, 7
k:	−13, 0	−7, 7
l:	0, 16	−18, 18
No. of standard reflections	1	1
Drift correction range	1·007–0·993	1·092–0·960
Total data measured	2124	4036
Data used in refinement	1763	2509
Parameters refined	375	565
R	0·0346	0·0786
wR [$w^{-1} = \sigma^2(F) + gF^2$]	0·0464	0·1033
S	0·812	2·05
Weighting-scheme parameter, *g*	0·00225	0·0028
Max. Δ/σ in last cycle	0·447	0·047
Final difference Fourier map peaks (e Å^{-3}) min.	−0·186	−0·33
max.	0·151	0·59

Table 2. *Atomic coordinates with equivalent isotropic temperature factors* (Å^2) *for* (1)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
C(1)	0·2868 (4)	0·0684 (3)	0·41933 (21)	0·0345 (17)
C(2)	0·4292 (4)	0·1133 (4)	0·48036 (25)	0·0417 (19)
C(3)	0·5127 (5)	0·2118 (4)	0·4522 (3)	0·0506 (23)
C(4)	0·4552 (5)	0·2637 (4)	0·3661 (3)	0·0511 (23)
C(5)	0·3180 (5)	0·2153 (4)	0·3053 (3)	0·0474 (22)
C(6)	0·2366 (4)	0·1165 (3)	0·33070 (22)	0·0362 (18)
C(7)	0·1042 (4)	0·0385 (3)	0·27228 (22)	0·0342 (17)
C(8)	0·0367 (4)	−0·0410 (3)	0·34827 (20)	0·0315 (17)
C(9)	−0·0478 (4)	0·0963 (3)	0·20637 (22)	0·0395 (19)
C(10)	0·0508 (5)	−0·0809 (4)	0·13452 (25)	0·0524 (23)
C(11)	0·2028 (4)	−0·0302 (4)	0·20212 (24)	0·0476 (21)
C(12)	−0·2191 (5)	0·1259 (3)	0·25040 (25)	0·0392 (18)
C(13)	−0·2884 (4)	0·0177 (3)	0·29328 (22)	0·0332 (17)
C(14)	−0·1489 (4)	−0·0137 (3)	0·37815 (20)	0·0286 (15)
C(15)	−0·2851 (4)	−0·0389 (4)	0·12059 (23)	0·0425 (19)
C(16)	−0·3169 (4)	−0·0740 (3)	0·21765 (22)	0·0351 (18)
C(17)	−0·3670 (5)	−0·1810 (4)	0·2340 (3)	0·0425 (20)
C(18)	−0·3903 (5)	−0·2238 (4)	0·3300 (3)	0·0496 (22)
C(19)	−0·1963 (5)	−0·1105 (3)	0·44216 (22)	0·0382 (19)
C(20)	−0·0387 (5)	−0·1310 (4)	0·52046 (23)	0·0451 (21)
C(21)	0·1336 (5)	−0·0622 (3)	0·51927 (22)	0·0388 (18)
N(1)	0·1756 (4)	−0·02682 (25)	0·43282 (18)	0·0343 (14)
N(2)	−0·0945 (4)	0·0108 (3)	0·12468 (18)	0·0446 (17)
O(1)	0·2310 (4)	−0·0396 (3)	0·59165 (16)	0·0539 (16)
O(2)	−0·2262 (3)	−0·21722 (23)	0·39394 (17)	0·0434 (14)
C(30)	−0·0441 (4)	0·0539 (3)	0·86621 (20)	0·0291 (15)
C(31)	0·1636 (4)	0·0615 (3)	0·86080 (23)	0·0343 (17)
C(32)	0·2395 (4)	−0·0544 (3)	0·83806 (22)	0·0311 (16)
C(33)	0·4466 (4)	−0·0522 (3)	0·84237 (21)	0·0309 (15)
O(301)	−0·1417 (3)	0·02389 (24)	0·79238 (15)	0·0433 (13)
O(302)	−0·0993 (3)	0·08118 (25)	0·94067 (15)	0·0435 (13)
O(31)	0·2595 (3)	0·1014 (3)	0·94513 (21)	0·0547 (16)
O(32)	0·1863 (3)	−0·13880 (22)	0·89899 (19)	0·0436 (13)
O(331)	0·5111 (3)	0·02727 (21)	0·79264 (16)	0·0386 (12)
O(332)	0·5372 (3)	−0·1215 (3)	0·88853 (20)	0·0579 (16)
O(1*W*)	0·1418 (4)	0·26924 (24)	0·05715 (19)	0·0461 (15)
O(2*W*)	0·6146 (4)	0·2085 (3)	0·98518 (24)	0·0704 (20)
O(3*W*)	0·8854 (6)	0·1512 (4)	0·6194 (3)	0·098 (3)

Table 3. *Atomic coordinates with equivalent isotropic temperature factors* (Å^2) *for* (2)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
C(1)	0·2709 (10)	−0·0308 (12)	−0·0685 (4)	0·045 (5)
C(2)	0·4100 (13)	−0·0494 (14)	−0·1233 (4)	0·063 (6)
C(3)	0·4897 (14)	−0·2132 (16)	−0·1294 (6)	0·071 (7)
C(4)	0·4347 (14)	−0·3466 (16)	−0·0848 (6)	0·070 (7)
C(5)	0·2981 (12)	−0·3253 (13)	−0·0300 (6)	0·063 (6)
C(6)	0·2170 (10)	−0·1677 (11)	−0·0236 (4)	0·047 (5)
C(7)	0·0738 (10)	−0·1097 (10)	0·0338 (4)	0·042 (5)
C(8)	0·0190 (10)	0·0676 (9)	0·0038 (4)	0·035 (4)
C(9)	−0·0799 (12)	−0·2321 (10)	0·0551 (4)	0·049 (5)
C(10)	−0·0162 (13)	−0·0765 (13)	0·1544 (5)	0·061 (6)
C(11)	0·1455 (12)	−0·0919 (12)	0·1023 (4)	0·051 (5)
C(12)	−0·2318 (11)	−0·2204 (10)	0·0124 (5)	0·048 (5)
C(13)	−0·3012 (10)	−0·0382 (10)	0·0118 (4)	0·043 (5)
C(14)	−0·1537 (11)	0·0723 (10)	−0·0289 (4)	0·043 (5)
C(15)	−0·3390 (12)	−0·1180 (13)	0·1401 (5)	0·056 (6)
C(16)	−0·3557 (10)	0·0151 (11)	0·0850 (5)	0·046 (5)
C(17)	−0·4206 (12)	0·1659 (13)	0·1015 (5)	0·058 (6)
C(18)	−0·4357 (13)	0·3027 (13)	0·0466 (6)	0·067 (6)
C(19)	−0·1920 (12)	0·2572 (11)	−0·0464 (5)	0·051 (5)
C(20)	−0·0205 (13)	0·3507 (11)	−0·0753 (5)	0·061 (6)
C(21)	0·1408 (13)	0·2441 (12)	−0·0962 (5)	0·055 (6)
N(1)	0·1665 (9)	0·1121 (8)	−0·0518 (3)	0·041 (4)
N(2)	−0·1509 (10)	−0·1919 (9)	0·1294 (4)	0·054 (5)
O(1)	0·2445 (10)	0·2733 (10)	−0·1508 (4)	0·079 (5)
O(2)	−0·2623 (9)	0·3553 (7)	0·0115 (4)	0·059 (4)
C(1′)	−0·1444 (11)	0·7462 (12)	−0·2852 (4)	0·045 (5)
C(2′)	−0·0477 (13)	0·7647 (16)	−0·2326 (5)	0·067 (7)
C(3′)	0·0260 (14)	0·9235 (17)	−0·2270 (5)	0·070 (7)
C(4′)	0·0096 (14)	1·0551 (16)	−0·2700 (6)	0·072 (7)
C(5′)	−0·0916 (12)	1·0393 (13)	−0·3267 (5)	0·065 (6)
C(6′)	−0·1635 (11)	0·8813 (11)	−0·3327 (4)	0·044 (5)
C(7′)	−0·2653 (10)	0·8196 (10)	−0·3895 (4)	0·039 (4)
C(8′)	−0·3426 (10)	0·6472 (9)	−0·3593 (4)	0·037 (4)
C(9′)	−0·4057 (12)	0·9432 (11)	−0·4114 (5)	0·054 (5)
C(10′)	−0·2633 (12)	0·7827 (14)	−0·5095 (5)	0·059 (6)
C(11′)	−0·1360 (12)	0·7992 (13)	−0·4566 (4)	0·055 (5)
C(12′)	−0·5840 (11)	0·9362 (10)	−0·3686 (5)	0·052 (5)
C(13′)	−0·6548 (10)	0·7511 (9)	−0·3686 (4)	0·041 (4)
C(14′)	−0·5383 (11)	0·6421 (10)	−0·3269 (4)	0·044 (4)
C(15′)	−0·5938 (13)	0·8303 (13)	−0·4978 (5)	0·064 (6)
C(16′)	−0·6514 (11)	0·6984 (11)	−0·4424 (4)	0·047 (5)
C(17′)	−0·7006 (13)	0·5441 (13)	−0·4549 (5)	0·063 (6)
C(18′)	−0·7533 (15)	0·4066 (13)	−0·4036 (7)	0·078 (7)
C(19′)	−0·5896 (12)	0·4543 (10)	−0·3092 (4)	0·048 (5)
C(20′)	−0·4486 (14)	0·3645 (12)	−0·2762 (5)	0·061 (6)
C(21′)	−0·2952 (12)	0·4720 (11)	−0·2567 (4)	0·051 (5)
N(1′)	−0·2360 (9)	0·6021 (9)	−0·3030 (3)	0·046 (4)
N(2′)	−0·4118 (10)	0·8991 (10)	−0·4879 (4)	0·060 (5)
O(1′)	−0·2304 (11)	0·4409 (10)	−0·2048 (4)	0·081 (5)
O(2′)	−0·6100 (9)	0·3579 (8)	−0·3680 (4)	0·065 (4)
C(30)	0·8065 (23)	0·5037 (21)	0·2567 (8)	0·110 (12)
C(31)	0·7484 (20)	0·3546 (14)	0·3053 (6)	0·089 (8)
C(32)	0·618 (3)	0·4082 (18)	0·3610 (7)	0·126 (13)
C(33)	0·549 (3)	0·2548 (17)	0·4141 (9)	0·124 (13)
O(301)	0·7931 (15)	0·4985 (11)	0·1977 (5)	0·109 (7)
O(302)	0·860 (3)	0·6328 (21)	0·2813 (7)	0·214 (16)
O(31)	0·6944 (24)	0·2171 (13)	0·2705 (5)	0·165 (11)
O(32)	0·461 (3)	0·4569 (24)	0·3327 (11)	0·272 (21)
O(331)	0·657 (3)	0·1935 (17)	0·4455 (6)	0·169 (11)
O(332)	0·400 (3)	0·206 (3)	0·4243 (11)	0·217 (18)
O(1*W*)	0·1140 (16)	0·3766 (15)	0·1162 (6)	0·133 (3)
O(2*W*)	0·6212 (19)	0·8692 (19)	0·3280 (8)	0·160 (4)
O(3*W*)	0·3573 (20)	0·5584 (19)	0·1877 (8)	0·165 (5)
O(4*W*)	0·8817 (23)	0·3501 (21)	0·5236 (9)	0·191 (6)
O(5*W*)	0·095 (3)	0·590 (3)	0·3789 (11)	0·218 (7)
O(6*W*)	0·237 (4)	0·199 (3)	0·2553 (14)	0·265 (10)

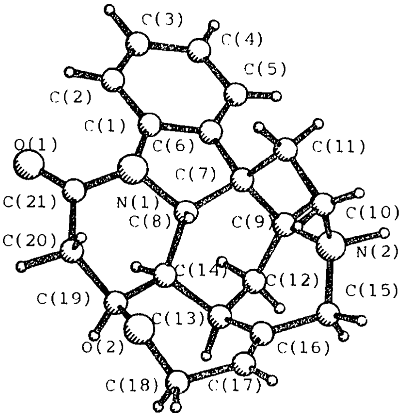


Fig. 1. Perspective drawing of strychnine and numbering scheme.

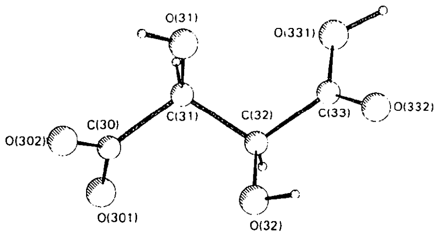


Fig. 2. (Bi)tartrate numbering scheme.

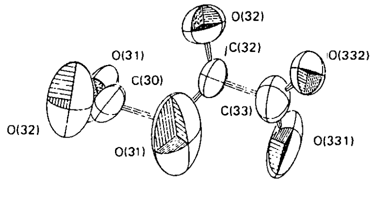


Fig. 3. Perspective drawing of the tartrate ion from (2), showing high thermal motion (50% probability ellipsoids).

standard methods, and anisotropic thermal parameters were refined for all non-H atoms except the water O atoms in (2). H atoms bonded to carbon were introduced in calculated positions, with idealized trigonal or tetrahedral symmetry and C—H = 1·08 Å. Positional parameters were refined for all H atoms bonded to oxygen in (1). Considerable difficulty was encountered in refining the positional parameters for the tartrate ion in (2), but bond-distance constraints, which were applied in the early stages of the refinement, were released for the final cycles. The large thermal parameters suggest that this ion, shown in Fig. 3, is partly disordered. The relatively high *R* value for (2) is attributed to this disorder. As there is little extended hydrogen bonding in this structure, it was not possible to locate or refine H atoms bonded to oxygen. The computer programs used in the work and not otherwise mentioned were: refinement and source of atomic scattering factors, *SHELX*76 (Sheldrick, 1976); molecular geometry, *CALC* (Gould & Taylor, 1983); diagrams, *PLUTO* (Motherwell, 1972) and *INTERACTIVE ORTEP* (Johnson, 1965; Mallinson, 1982). Positional parameters for non-H atoms are shown in

Table 4. *Selected strychnine bond lengths* (Å), *angles* (°) *and torsion angles* (°)

	(1)	(2)	(2′)
C(9)–N(2)	1·545 (5)	1·521 (11)	1·559 (12)
C(10)–N(2)	1·512 (5)	1·515 (12)	1·468 (13)
C(15)–N(2)	1·515 (5)	1·524 (12)	1·525 (13)
C(19)–C(20)–C(21)	118·3 (3)	117·0 (8)	117·9 (8)
C(20)–C(21)–N(1)	117·1 (3)	115·4 (8)	115·3 (7)
C(20)–C(21)–O(1)	121·2 (3)	122·4 (9)	121·9 (8)
N(1)–C(21)–O(1)	121·7 (3)	122·1 (9)	122·8 (8)
C(14)–C(19)–C(20)–C(21)	−3·6 (4)	13·5 (11)	8·0 (11)
O(2)–C(19)–C(20)–C(21)	119·1 (3)	136·6 (8)	131·6 (8)
C(19)–C(20)–C(21)–N(1)	−29·0 (5)	−43·0 (12)	−38·4 (12)
C(19)–C(20)–C(21)–O(1)	151·3 (4)	136·6 (10)	143·0 (9)
C(20)–C(21)–N(1)–C(1)	156·1 (3)	159·7 (8)	157·7 (8)
C(20)–C(21)–N(1)–C(8)	9·9 (4)	11·5 (11)	10·9 (11)

Table 5. (*Bi*)*tartrate bond lengths* (Å), *angles* (°) *and torsion angles* (°)

	(1)	(2)		(1)	(2)		(1)
C(30)–C(31)	1·546 (4)	1·505 (21)	C(32)–C(33)	1·521 (4)	1·60 (3)	C(32)–H(32C)	0·96 (4)
C(30)–O(301)	1·252 (4)	1·178 (20)	C(32)–O(32)	1·405 (4)	1·42 (3)	O(31)–H(31O)	0·81 (4)
C(30)–O(302)	1·228 (4)	1·236 (24)	C(33)–O(331)	1·297 (4)	1·181 (25)	O(32)–H(32O)	0·90 (4)
C(31)–C(32)	1·518 (5)	1·435 (22)	C(33)–O(332)	1·197 (4)	1·19 (3)	O(331)–H(331)	0·94 (4)
C(31)–O(31)	1·401 (4)	1·393 (18)	C(31)–H(31C)	1·04 (4)			
C(31)–C(30)–O(301)	116·4 (3)	120·3 (14)	C(31)–C(32)–O(32)	111·0 (3)	107·7 (15)	O(31)–C(31)–H(31C)	110·1 (22)
C(31)–C(30)–O(302)	117·7 (3)	118·1 (15)	C(33)–C(32)–O(32)	110·0 (3)	102·9 (15)	C(31)–C(32)–H(32C)	108·6 (25)
O(301)–C(30)–O(302)	125·9 (3)	121·6 (17)	C(32)–C(33)–O(331)	115·1 (3)	115·7 (17)	C(33)–C(32)–H(32C)	103·9 (25)
C(30)–C(31)–C(32)	110·7 (3)	110·1 (12)	C(32)–C(33)–O(332)	120·0 (3)	123·9 (19)	O(32)–C(32)–H(32C)	111·3 (25)
C(30)–C(31)–O(31)	112·0 (3)	111·9 (12)	O(331)–C(33)–O(332)	124·9 (3)	120·4 (21)	C(31)–O(31)–H(31O)	117 (3)
C(32)–C(31)–O(31)	108·8 (3)	114·0 (12)	C(30)–C(31)–H(31C)	103·5 (22)		C(32)–O(32)–H(32O)	110 (3)
C(31)–C(32)–C(33)	111·8 (3)	112·6 (14)	C(32)–C(31)–H(31C)	111·7 (22)		C(33)–O(331)–H(331)	111·8 (24)
O(301)–C(30)–C(31)–C(32)	−61·5 (4)	−123·5 (16)	O(32)–C(32)–C(33)–O(331)	−176·3 (3)	−178·9 (18)	C(32)–C(31)–O(31)–H(31O)	−146 (3)
O(301)–C(30)–C(31)–O(31)	176·8 (3)	4·4 (21)	O(32)–C(32)–C(33)–O(332)	4·5 (4)	−1 (3)	H(31C)–C(31)–O(31)–H(31O)	90 (4)
O(302)–C(30)–C(31)–C(32)	120·8 (3)	53·2 (20)	O(301)–C(30)–C(31)–H(31C)	58·3 (23)		H(32C)–C(32)–C(33)–O(331)	64 (3)
O(302)–C(30)–C(31)–O(31)	−0·8 (4)	−178·9 (15)	O(302)–C(30)–C(31)–H(31C)	−119·4 (23)		H(32C)–C(32)–C(33)–O(332)	−114 (3)
C(30)–C(31)–C(32)–C(33)	−174·05 (25)	179·5 (13)	C(30)–C(31)–C(32)–H(32C)	71 (3)		C(31)–C(32)–O(32)–H(320)	−122 (3)
C(30)–C(31)–C(32)–O(32)	−50·8 (3)	66·8 (17)	O(31)–C(31)–C(32)–H(32C)	−164 (3)		C(33)–C(32)–O(32)–H(320)	2 (3)
O(31)–C(31)–C(32)–C(33)	−50·6 (3)	52·8 (18)	H(31C)–C(31)–C(32)–C(33)	71·2 (24)		H(32C)–C(32)–O(32)–H(320)	116 (4)
O(31)–C(31)–C(32)–O(32)	72·6 (3)	−59·9 (18)	H(31C)–C(31)–C(32)–O(32)	−165·6 (24)		C(32)–C(33)–O(331)–H(331)	−170 (3)
C(31)–C(32)–C(33)–O(331)	−52·5 (4)	65·5 (22)	H(31C)–C(31)–C(32)–H(32C)	−42 (4)		O(332)–C(33)–O(331)–H(331)	8 (3)
C(31)–C(32)–C(33)–O(332)	128·3 (3)	−116·9 (22)	C(30)–C(31)–O(31)–H(310)	−23 (3)			

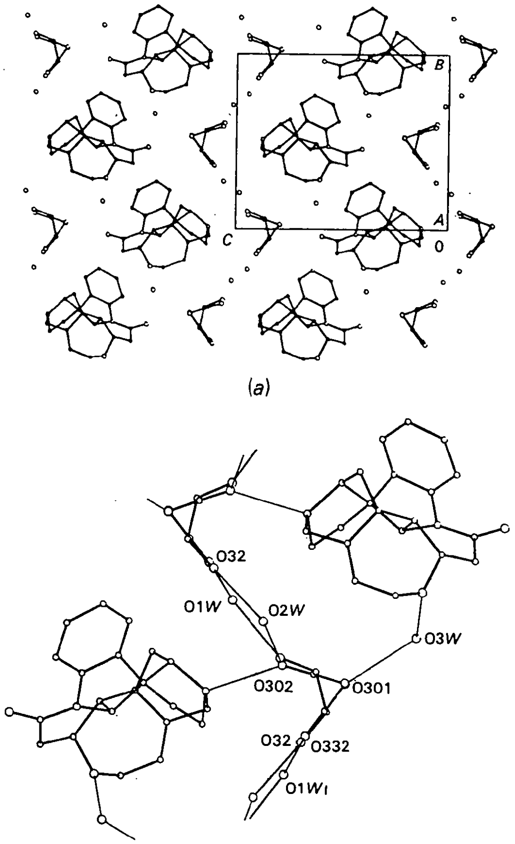
Tables 2 (1) and 3 (2).* Plots of the strychnine and tartrate moieties are shown in Figs. 1 and 2 together with the numbering system employed.

Discussion. Selected bond lengths, angles and torsion angles are tabulated in Tables 4 and 5. The three independent strychnine molecules are virtually indistinguishable. This is not surprising, as strychnine is an essentially rigid molecule, and its conformation differs little from one structure to another. Thus, the non-hydrogen skeleton of the strychnine ion from (1) may be superimposed on the molecules of (2) with an r.m.s. deviation of 0·155 and 0·099 Å respectively. The largest deviations occur in the most flexible part of the ion, the amide ring, and are reflected in the torsion angle C(19)—C(20)—C(21)—N(1), which has a range of 14° in (1) and (2). The angle C(19)—C(20)—C(21) is very large for an angle at an sp^3 carbon, being an average of 1·8° larger than the angle C(20)—C(21)—N(1), at an sp^2 carbon. The strychnine ion in (1) displays another anomaly also noted previously (Gould, Kelly & Walkinshaw, 1985) that C(9)—N(2) is 0·031 (7) Å longer than C(10)—N(2) and C(15)—N(2). A similar difference may also be seen in the two ions in (2), although the lower precision makes it less significant.

The disorder associated with the tartrate ion in (2) makes a detailed comparison with the geometry of the bitartrate ion in (1) impossible. A connectivity search of the Cambridge Structural Database (CSD) (Allen *et al.*, 1979) for compounds containing the tartrate ion not complexed to metals gave a total of 31 structures with 32 unique tartaric acid fragments.* The *trans* conformation of the two carboxylate groups about the central C—C bond [*i.e.* with the torsion C(30)—C(31)—C(32)—C(33) within ±10° of 180°] is found in 28 of these fragments as well as in both (1) and (2). The planes of the carboxylic acid groups tend to be coplanar with their vicinal hydroxyl group. In the 32 fragments there are 64 unique torsions of the type O(301)—C(30)—C(31)—O(31), and 39 of these torsions are less than 10°.

Molecular packing

A major reason for studying these salts is to observe the packing of the attached ions, and to elucidate factors affecting the choice of types of strychnine packing in any salt. Packing diagrams for the two structures are shown in Figs. 4 and 5. The two packing arrangements are superficially similar, each consisting of infinite bilayers of alkaloid separated by layers of counterion and water. In (1), the bitartrate ions are strongly linked both by direct hydrogen bonds and by hydrogen bonds *via* the water molecules [water bond lengths and angles in (1) are given in Table 6]. The direct hydrogen bonds form infinite chains in the *x* direction, and these chains are joined into sheets by the water molecules. The sheets link to strychnine ions *via* a 'salt bridge' hydrogen bond between the charged acid function of the bitartrate and the protonated N(2) of the alkaloid. Another linkage occurs between a water hydrogen and the amide oxygen [O(1)] of the strychnine ion.



D···*A*		*D*···*A* (Å)	H···*A* (Å)	∠*D*—H···*A* (°)
N(2)	O(302)	2·752 (4)	1·707	163·3
O(331)	O(301)	2·561 (3)	1·63 (4)	170 (4)
O(1*W*)	O(332)	2·719 (4)	1·94 (5)	162 (5)
O(2*W*)	O(302)	2·722 (4)	1·96 (4)	153 (4)
O(1*W*)	O(32)	2·790 (4)	1·90 (4)	172 (4)
O(2*W*)	O(31)	2·896 (4)	2·08 (4)	174 (4)
O(31)	O(1*W*)	2·743 (4)	1·97 (4)	159 (4)
O(32)	O(2*W*)	2·738 (4)	1·92 (4)	149 (4)
O(3*W*)	O(301)	2·916 (5)	2·04 (4)	156 (4)
O(3*W*)	O(2)	2·974 (5)	2·11 (4)	155 (4)

(*b*)

Fig. 4. (*a*) Packing diagram of (1), *a*-axis projection; (*b*) hydrogen bonding.

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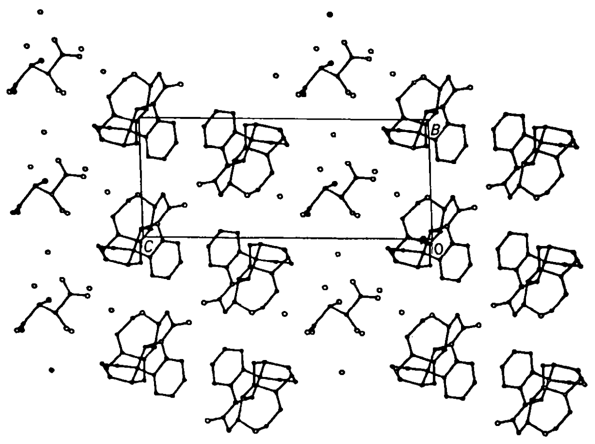
* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, full lists of strychnine bond lengths, angles and torsion angles and CSD REFCODES of tartrate compounds used in structural comparison have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44282 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

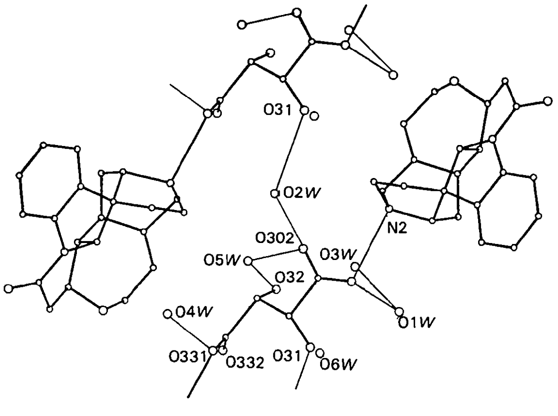
In contrast to this well defined pattern, the hydrogen bonding in (2) is less striking, as the sheets are held together by water molecules in both the x and the y directions, and the only hydrogen-bonded contact of the alkaloid with this sheet is the 'salt bridge' bond.

The strychnine layers are themselves held together by many weak non-bonded interactions, of which the most important are listed in Table 7. No single strong contact can be identified that is responsible for the layer packing. Although there are differences between the intralayer forces in the two structures, there is one important common feature. This is the short contact between a methine hydrogen and the aromatic ring of the adjacent strychnine ion, separated from the first by a unit-cell translation in **a**. These contacts are best indicated by H(13)—C(6) in (1), and C(2)—H(131) and C(2′)—H(131′) in the two independent strychnine ions of (2). Thus, the two layer types have a common substructure of strychnine columns.

There are two distinct alkaloid packing types in the present structures. That exhibited by (1) is also seen in the salt of strychnine and *N*-benzoyl-L-alanine (Gould *et al.*, 1985), and can be described as a grooved bilayer. Interaction of counterions with this bilayer occurs mainly by hydrogen bonding, with the bilayer presenting two different hydrogen-bonding sites to the counterion/water space. In (2), the alkaloid packing is similar to that seen in the mineral-acid salts (Cleasby, Gould, Moulden & Walkinshaw, 1981), and the surface presented to the counterion/water space is less well featured. In this type of alkaloid packing, the amide oxygen is kept inside the bilayer, and so is unavailable for hydrogen bonding. The disorder in the tartrate ion and the loosely ordered water about it suggests that either type of tartrate could fit into this type of packing, giving it low chiral specificity.



(a)



D···*A*		*D*···*A* (Å)	H···*A* (Å)	∠*D*—H···*A* (°)
N(2′)	O(331)	2·626 (17)	1·568	165·6
N(2)	O(301)	2·735 (13)	1·662	171·1
O(302)	O(2*W*)	2·676 (23)		
O(302)	O(5*W*)	2·80 (3)		
O(301)	O(1*W*)	2·885 (16)		
O(31)	O(2*W*)	2·940 (20)		
O(32)	O(5*W*)	2·99 (3)		
O(1*W*)	O(3*W*)	2·887 (19)		

(b)

Fig. 5. (*a*) Packing diagram of (2), *a*-axis projection; (*b*) hydrogen bonding.

Table 6. *Water bond lengths* (Å) *and angles* (°) *from* (1)

O(1*W*)–H(11*W*)	0·90 (4)	O(1*W*)–H(12*W*)	0·81 (5)
O(2*W*)–H(21*W*)	0·83 (4)	O(2*W*)–H(22*W*)	0·82 (4)
O(3*W*)–H(31*W*)	0·93 (4)	O(3*W*)–H(32*W*)	0·92 (4)
H(11*W*)–O(1*W*)–H(12*W*)	105 (4)	H(31*W*)–O(3*W*)–H(32*W*)	88 (4)
H(21*W*)–O(2*W*)–H(22*W*)	104 (4)		

Table 7. *Intermolecular contacts* (Å)

Structure (1)			
Intermolecular C—C distances less than 3·6 Å			
C(2)–C(18^{i})	3·356 (6)	C(11)–C(16^{ii})	3·559 (5)
C(3)–C(18^{i})	3·430 (6)	C(3)–C(19^{i})	3·595 (5)
C(4)–C(12^{ii})	3·477 (5)		
Intermolecular C—O distances less than 3·4 Å			
C(2)–O(2^{i})	3·174 (4)	C(3)–O(2^{i})	3·337 (5)
C(4)–O(1^{iii})	3·269 (5)	C(15)–O(302^{v})	3·367 (4)
C(33)–O(301^{ii})	3·325 (4)	C(9)–O(1*W*^{vi})	3·368 (4)
C(30)–O(331^{iv})	3·333 (4)		
Structure (2)			
Intermolecular C—C distances less than 3·6 Å			
C(4′)–C(12′^{ii})	3·527 (14)	C(4)–C(12^{ii})	3·547 (14)
Intermolecular C—N distances less than 3·6 Å			
C(33)–N(2′^{vii})	3·362 (21)	C(30)–N(2^{viii})	3·369 (18)
Intermolecular C—O distances less than 3·4 Å			
C(20′)–O(1^{iv})	3·189 (13)	C(9′)–O(331^{ix})	3·338 (17)
C(20)–O(1′^{vi})	3·237 (12)	C(10)–O(302^{xii})	3·342 (20)
C(15′)–O(332^{ix})	3·273 (23)	C(12)–O(2^{xiii})	3·344 (11)
C(12′)–O(2′^{x})	3·318 (11)	C(15)–O(301^{xii})	3·371 (14)
C(18′)–O(4*W*^{xi})	3·329 (21)	C(10′)–O(331^{ix})	3·384 (18)

Symmetry code: (i) $-x, \frac{1}{2}+y, 1-z$; (ii) $1+x, y, z$; (iii) $1-x, \frac{1}{2}+y, 1-z$; (iv) $-1+x, y, z$; (v) $x, y, -1+z$; (vi) x, y, z; (vii) $1+x, -1+y, 1+z$; (viii) $1+x, 1+y, z$; (ix) $-1+x, 1+y, -1+z$; (x) $x, 1+y, z$; (xi) $-2+x, y, -1+z$; (xii) $-1+x, -1+y, z$; (xiii) $x, -1+y, z$.

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Acta Cryst. (1987). C**43**, 2410–2412

5-Methyl-2,8-dithia-1,3,7-triphosphatricyclo$[3.3.1.0^{3,7}]$nonane 1-Sulfide

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Abstract. $C_5H_9P_3S_3$, $M_r = 258{\cdot}23$, monoclinic, $A2/a$, $a = 12{\cdot}027\ (5)$, $b = 7{\cdot}785\ (1)$, $c = 22{\cdot}762\ (14)$ Å, $\beta = 102{\cdot}44\ (7)°$, $V = 2081\ (3)$ Å^3, $Z = 8$, $D_x = 1{\cdot}65$ g cm^{-3}, λ(Mo $K\alpha$) = 0·70926 Å, $\mu = 10{\cdot}81$ cm^{-1}, $F(000) = 1056$, $T = 295\ (2)$ K, $R = 0{\cdot}025$ for 1843 unique reflections. The molecule consists of a folded five-membered P_3S_2 ring with one exocyclic S atom; the ring is capped by the organic group $CH_3C(CH_2)_3$ which is bonded to the P atoms. A short intermolecular P···P distance of 3·033 (1) Å is found in the crystal structure.

Introduction. The title compound $CH_3C(CH_2P)_3S_2(S)$ was prepared by Ellermann, Demuth & Bauer (1986). These authors postulated a five-membered P_3S_2 ring with an exocylic S atom by spectroscopic methods. The X-ray structure of the analogous arsenic compound $CH_3C(CH_2As)_3S_3$ (Ellermann, Lietz, Merbach, Thiele & Zoubek, 1979) has a six-membered As_3S_3 ring. An X-ray analysis has been performed to confirm the proposed unusual structure of $CH_3C(CH_2P)_3S_2(S)$.

Experimental. Recrystallization from tetrahydrofuran gave colourless crystals; spherical crystal with $r = 0{\cdot}18\ (2)$ mm used for X-ray analysis; Philips PW 1100 diffractometer (graphite monochromator, Mo $K\alpha$ radiation); ω–2θ scan mode, scan width $(1{\cdot}000 + 0{\cdot}345\tan\theta)°$, profile analysis with a modified Lehmann & Larsen (1974) procedure; lattice parameters by least-squares refinement (*GIVER*: Krogmann, 1966) on the basis of 48 reflections with $21 \leq 2\theta \leq 29°$; spherical absorption correction applied with $\mu R = 0{\cdot}20\ (2)$, absorption correction factor $A = 1{\cdot}33\ (3)$; index range h −14/13, k 0/9, l 0/26; six standard reflections (intensity variation 0·3%); 7362 reflections measured, $(\sin\theta_{\max})/\lambda = 0{\cdot}60$ Å^{-1}, after averaging symmetrical equivalents with $I > 3\sigma(I)$ 1843 reflections remained, $R_{\rm int} = 0{\cdot}01$. Structure solved by direct methods, subsequent Fourier map revealed all C atoms and all H atoms could be located from a difference Fourier map; final refinement by blocked least-squares methods (based on F) of scale factor, coordinates and anisotropic temperature parameters (H atoms isotropic). $R = 0{\cdot}025$, $wR = 0{\cdot}022$, $w = 1/\sigma^2(F_o)$, $S =$

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