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# The crystal structure of strychnidine succinate shows similar self-assembly to strychnine salts

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In the crystal structure of strychnidine succinate, $C_{21}H_{25}N_2O^+\cdot C_4H_5O_4^-$, strychnidinium bilayers are separated by layers composed of extended carboxylic acid O—H···O hydrogen-bonded chains. The significantly different C—O distances of the carboxylate groups of the succinate anion allow the negatively charged O atoms to be identified.

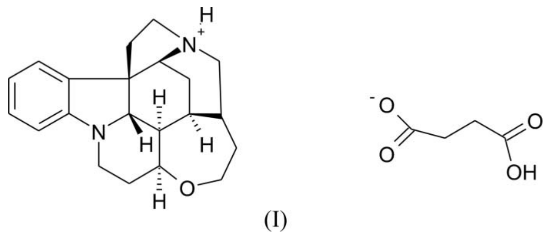
**Key indicators**

Single-crystal X-ray study
*T* = 100 K
Mean *σ*(C–C) = 0.003 Å
*R* factor = 0.033
*wR* factor = 0.077
Data-to-parameter ratio = 10.3

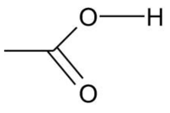
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

## Comment

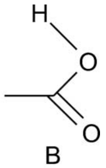
The structurally related, naturally occurring alkaloids strychnine and brucine have become known as powerful resolving agents, originating from the work of Fisher (1899). Surprisingly, there are no data available on separating procedures involving strychnine and brucine derivatives, and neither have many structures of such compounds been published. Recently, we turned our attention to the possibility of utilizing strychnidine, a reduced form of strychnine, as a chiral adjuvant. Strychnidine is a readily available, less toxic, equally stable and an enantiomerically pure derivative of strychnine (Zwicker & Robinson, 1942; Clemo *et al.*, 1927). We have determined the crystal structure of strychnidine succinate, (I), and a view of the asymmetric unit is shown in Fig. 1.



(I)



A



B

The title compound forms crystals resembling those of some strychnine salt crystals. A reason could be that the title structure, like some strychnine salts, is built from alternating cation bilayers and anion monolayers. In (I), the succinic acid anions, where the H atom of one of the carboxyl groups is dissociated, form one-dimensional O—H···O hydrogen-bonded chains in the *a*-axis direction. The strychnidine molecules pack with the hydrophobic parts of the molecules oriented back to back and the protonated amine N atoms are N—H···O hydrogen bonded to carboxylate O atoms (Table 2

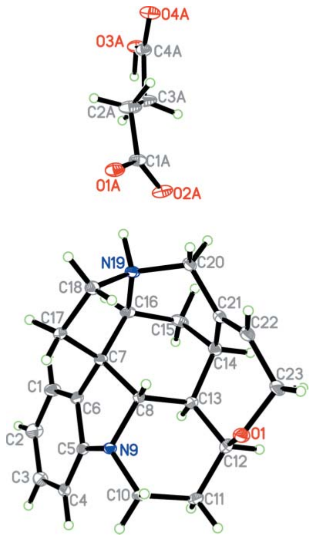


**Figure 1**
The asymmetric unit of (I), showing 30% probability displacement ellipsoids for non-H atoms and H atoms drawn as small spheres.

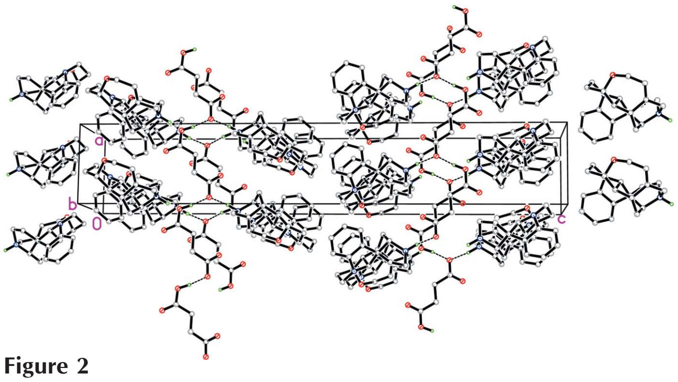


**Figure 2**
A packing diagram of (I), showing hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

and Fig. 2). Similar types of crystal structures were observed previously not only for strychnine–carboxylic acid salts (Gould & Walkinshaw, 1984; Gould *et al.*, 1985; Białońska & Ciunik, 2004; Gould *et al.*, 1987), but also for inorganic salts of strychnine (Białońska & Ciunik, 2005).

We have examined the geometry of the carboxylic acid fragment in (I) with respect to carboxylic acid structures in the Cambridge Structural Database (Version 5.26; Allen, 2002). A search found 2050 hits for carboxylic acid structures in which hydrogen bonds are formed between a carboxyl group and a carbonyl O atom from a second carboxyl group. Furthermore there are 853 structures in which a molecule contains both a carboxyl and a carboxylate group. In some of these structures the double and the single bonds in the carboxylate groups can be identified and hence the negatively charged O atom (see, for example, Maurin *et al.*, 2003). Table 1 lists the geometries of the carboxyl and carboxylate groups in (I) and shows that the C—O bond lengths in the carboxyl group (C4*A*—O3*A* and C4*A*—O4*A*) have normal values for C—O(H) and C=O bonds, respectively. We have compared the bond lengths in the carboxylate group with similar structures found in the Cambridge Structural Database. The related structures all have the same type of carboxyl group which is hydrogen bonded to a carboxylate anion (see Fig. 3). In most structures long C—O bond lengths are between 1.250 and 1.275 Å, whereas short C—O distances are between 1.215 and 1.260 Å. C—O bond distances exceeding 1.285 Å are uncommon. In this respect, the observed lengths of 1.298 (2) and 1.230 (2) Å in the title compound represent a rare situation where the descriptions ‘single’ and ‘double’ bonds are justified.

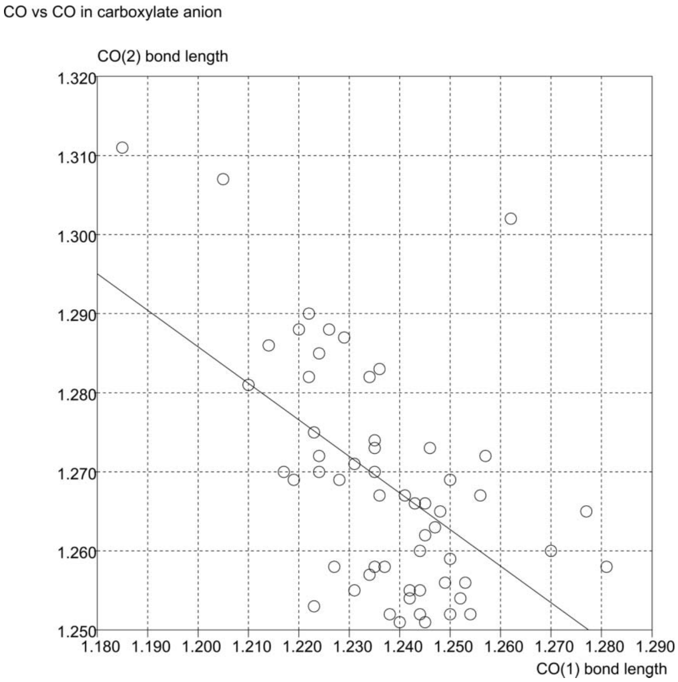


**Figure 3**
A graph illustrating the relationship between ‘single’ [CO(2)] and ‘double’ [CO(1)] bond lengths in structures with carboxylate anionic groups.

## Experimental

Strychnidine and succinic acid were dissolved in *n*-propanol in a 1:1 molar ratio. Crystals of the strychnidine succinate were obtained after slow evaporation of the solvent.

### *Crystal data*

$C_{21}H_{25}N_2O^+\cdot C_4H_5O_4^-$
$M_r = 438.51$
Orthorhombic, $P2_12_12_1$
$a = 6.487$ (2) Å
$b = 8.314$ (2) Å
$c = 39.165$ (6) Å
$V = 2112.3$ (9) Å$^3$
$Z = 4$
$D_x = 1.379$ Mg m$^{-3}$

Cu $K\alpha$ radiation
Cell parameters from 8322 reflections
$\theta = 4.5$–74.8°
$\mu = 0.78$ mm$^{-1}$
$T = 100$ (2) K
Plate, colourless
0.18 × 0.10 × 0.02 mm

### *Data collection*

Xcalibur PX kappa-geometry diffractometer with Onyx CCD detector
$\omega$ and $\varphi$ scans
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2003)
$T_{\min} = 0.842$, $T_{\max} = 0.988$

13198 measured reflections
3073 independent reflections
2730 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 68.8°$
$h = -6 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -42 \rightarrow 38$

*Acta Cryst.* (2006). E**62**, o694–o696 Maurin *et al.* • $C_{21}H_{25}N_2O^+\cdot C_4H_5O_4^-$ **o695**

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.077$
$S = 1.00$
3073 reflections
298 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.050P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.21$ e Å$^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0030 (3)
Absolute structure: Flack (1983), 1182 Friedel pairs
Flack parameter: 0.0 (2)

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| O1*A*—C1*A* | 1.298 (2) | O3*A*—C4*A* | 1.324 (2) |
| O2*A*—C1*A* | 1.230 (2) | O4*A*—C4*A* | 1.213 (2) |

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N19—H19···O1*A* | 1.02 (2) | 1.69 (2) | 2.700 (2) | 170.0 (19) |
| N19—H19···O2*A* | 1.02 (2) | 2.54 (2) | 3.225 (2) | 123.5 (16) |
| O3*A*—H3*A*···O1*A*$^{i}$ | 0.98 (3) | 1.63 (3) | 2.598 (2) | 173 (2) |

Symmetry code: (i) $x + 1, y, z$.

H atoms bonded to C atoms were placed in calculated positions with C—H = 0.95–1.00 Å. They were included in the refinement with $U_{iso}(H) = 1.2U_{eq}(C)$. The H atom bonded to an O atom was refined isotropically.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 1999); software used to prepare material for publication: *SHELXL97*.

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**o696** Maurin *et al.* • $C_{21}H_{25}N_2O^+ \cdot C_4H_5O_4^-$ *Acta Cryst.* (2006). E**62**, o694–o696