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People's Republic of ChinaCorrespondence e-mail: suncuirong@zju.edu.cnDicatharanthinium (2*R*,3*R*)-tartrate
trihydrate

The title salt, bis(methyl 3,4-didehydroibogamin-6-ium-18-carboxylate) (2*R*,3*R*)-tartrate trihydrate, $2C_{21}H_{25}N_2O_2^+ \cdot C_4H_4O_6^{2-} \cdot 3H_2O$, contains two catharanthinium cations, a (2*R*,3*R*)-tartrate anion and three water solvent molecules. The cation contains an indole ring system with a planar conformation and a seven-membered nitrogen-containing ring with a distorted chair conformation. A network of O—H···O and N—H···O intermolecular hydrogen bonds stabilizes the crystal packing.

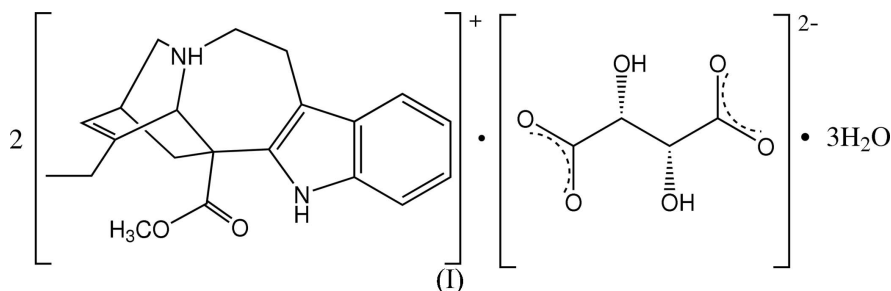
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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(C-C) = 0.007\text{ \AA}$
 R factor = 0.063
 wR factor = 0.136
Data-to-parameter ratio = 7.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Comment

Catharanthine is an important member of the iboga class of indole alkaloids (Gorman *et al.*, 1959). It is a chemical and presumed biological precursor of the antitumor alkaloids vinblastine and vincristine, two effective anticancer agents used in the treatment of a number of human cancers (Neuss & Neuss, 1990). Its synthesis has been widely investigated (Reding *et al.*, 1999; Büchi *et al.*, 1970).



The catharanthinium cation of the title salt, (I), contains an indole ring system and a seven-membered nitrogen-containing ring (Fig. 1). The structure of this cation is similar to that of ibogamine, except for the incorporation of the $COOCH_3$ group in position C9 and the double-bond character of $C16=C17$ (Soriano-García *et al.*, 1988). The indole ring system is planar and the seven-membered ring adopts a distorted chair conformation, which has been observed in the X-ray crystallographic study of ibogamine (Soriano-García *et al.*, 1988). The fused six-membered rings in the cations ($C13-C15/N2/C10/C9$ and $C14/C16/C17/C10/N2/C15$, or $C34-C36/N4/C31/C30$ and $C35/C37/C38/C31/N4/C36$) adopt boat conformations. There are three chiral centres ($C9/C10/C14$ and $C30/C31/C35$) in the catharanthinium cations, and the assigned absolute configurations are $9R,10S,14S$ and $30R,31S,35S$, according to the absolute configuration of the starting (2*R*,3*R*)-tartaric acid. Selected bond lengths and angles are listed in Table 1.

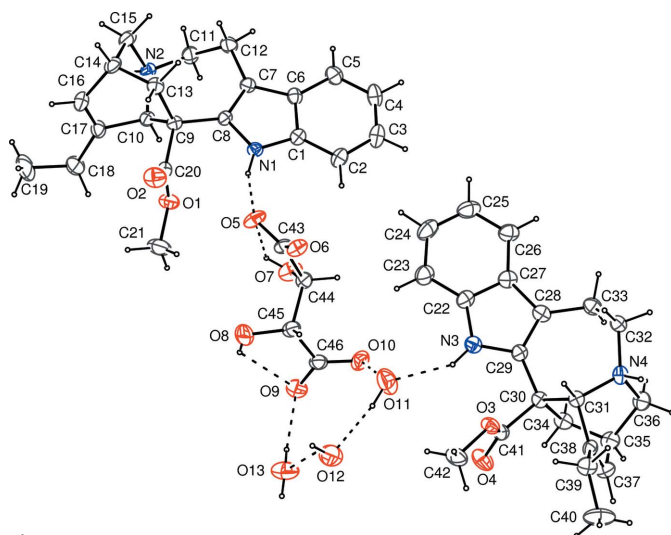


Figure 1
The asymmetric unit of the title salt, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

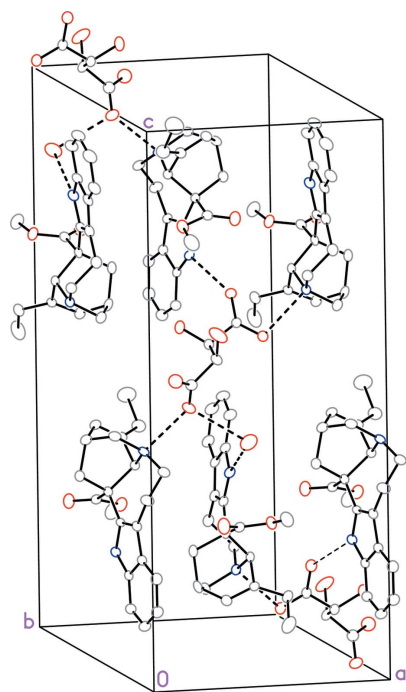


Figure 2
The packing of (I), showing O—H...O and N—H...O hydrogen-bond interactions as dashed lines. H atoms have been omitted.

The packing of the salt in the crystal structure is illustrated in Fig. 2; it is stabilized by a network of intermolecular O—H...O and N—H...O interactions (Table 2).

Experimental

Catharanthine was isolated from *Catharanthus roseus* G. Don, and it was dissolved with equimolar (2*R*,3*R*)-tartaric acid in ethanol (containing 5% water). Single crystals suitable for X-ray structure analysis were obtained by slow evaporation of the solution at room temperature.

Crystal data

$2C_{21}H_{25}N_2O_2^+ \cdot C_4H_4O_6^{2-} \cdot 3H_2O$
 $M_r = 876.98$
 Orthorhombic, $P2_12_12_1$
 $a = 10.1147$ (4) Å
 $b = 18.8215$ (8) Å
 $c = 23.4907$ (9) Å
 $V = 4472.0$ (3) Å³
 $Z = 4$
 $D_x = 1.303$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 3507 reflections
 $\theta = 2.2\text{--}24.1^\circ$
 $\mu = 0.10$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.31 \times 0.27 \times 0.23$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.965$, $T_{\max} = 0.971$
 24131 measured reflections

4516 independent reflections
 3899 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\max} = 25.2^\circ$
 $h = -12 \rightarrow 7$
 $k = -22 \rightarrow 22$
 $l = -27 \rightarrow 28$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.136$
 $S = 1.19$
 4516 reflections
 574 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 1.3486P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

N2—C11	1.497 (6)	C9—C10	1.560 (6)
N2—C15	1.506 (6)	C11—C12	1.505 (6)
N2—C10	1.518 (5)	C14—C16	1.502 (7)
C8—C9	1.523 (5)	C16—C17	1.327 (7)
C9—C20	1.538 (6)	C17—C18	1.501 (7)
C11—N2—C15	114.9 (4)	N2—C11—C12	116.5 (4)
C11—N2—C10	117.6 (3)	C7—C12—C11	114.4 (4)
C15—N2—C10	110.7 (3)	C16—C14—C15	106.9 (4)
C8—C9—C20	109.2 (3)	C15—C14—C13	107.3 (4)
C20—C9—C13	108.7 (4)	C17—C16—C14	115.4 (4)
C17—C10—N2	105.8 (3)	C16—C17—C10	111.5 (4)
N2—C10—C9	111.4 (3)	C18—C17—C10	119.6 (4)
N1—C8—C9—C13	110.8 (4)	C13—C9—C10—N2	53.2 (4)
C8—C9—C10—N2	-70.2 (4)	C9—C10—C17—C16	58.6 (5)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A...O5	0.86	2.02	2.821 (5)	155
N2—H2...O10 ⁱ	0.91	1.76	2.653 (5)	167
N3—H3A...O11	0.86	1.99	2.773 (5)	151
N4—H4...O6 ⁱⁱ	0.91	1.82	2.713 (4)	166
O7—H7...O5	0.82	2.19	2.623 (4)	113
O8—H8...O9	0.82	2.15	2.631 (7)	117
O11—H11C...O10	0.85	2.02	2.862 (6)	172
O11—H11D...O12	0.86	1.89	2.726 (5)	165
O12—H12C...O9 ⁱⁱⁱ	0.85	1.89	2.720 (6)	164
O12—H12D...O13	0.86	1.96	2.760 (7)	155
O13—H13C...O6 ^{iv}	0.83	1.97	2.781 (5)	166
O13—H13D...O9	0.84	2.04	2.854 (5)	164

Symmetry codes: (i) $-x + \frac{1}{2}, -y + 1, z + \frac{1}{2}$; (ii) $-x + \frac{3}{2}, -y + 1, z - \frac{1}{2}$; (iii) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (iv) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$.

H atoms on water molecules were located in a difference Fourier map and refined as riding in their as-found positions, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. Other H atoms were placed in calculated positions, with C–H = 0.93 (aromatic), 0.96 (methyl), 0.97 (methylene) or 0.98 Å (methine) and N–H = 0.86 (conjugated) or 0.91 Å (quaternary amine), and were refined as riding, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl})$ or $1.2U_{\text{eq}}(\text{other C or N})$. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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