Cinchonidinium (*R,R*)-tartrate monohydrate

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Key indicators

Single-crystal X-ray study
$T = 122$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.000
Data-to-parameter ratio = 10.9

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

Two symmetry-related cinchonidinium ions of the title compound, $C_{19}H_{23}N_2O^+ \cdot 0.5C_4H_4O_6^{2-} \cdot H_2O$, are linked through hydrogen bonds, with a tartrate ion positioned on the twofold axis.

Comment

As part of our investigations of how the packing of salts of optically active tartaric acid are influenced by the nature of the counter-ion, we have investigated diastereomeric salts formed by cinchona alkaloids and optically active tartaric acid. We report here the structure of (18*R*,19*S*,21*S*,25*S*)-cinchonidinium (2*R*,3*R*)-tartrate monohydrate, (I).

(I)
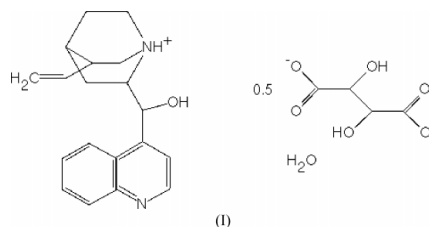
The tartrate ion is located on the crystallographic twofold axis. A hydrogen bond is formed between the carboxylate group of the tartrate ion and the quinuclidine group of the cinchonidinium ion. The packing resembles the arrangement found in other salts of the cinchona alkaloids (Gjerløv & Larsen, 1997), in contrast with a previously determined structure of a salt with a cinchona alkaloid and optically active tartaric acid (Ryttersgaard & Larsen, 1998).

Experimental

Crystals of (I) were obtained by mixing ethanol solutions of cinchonine and (*R,R*)-tartaric acid in a 1:3 molar ratio.

Crystal data

$C_{19}H_{23}N_2O^+ \cdot 0.5C_4H_4O_6^{2-} \cdot H_2O$	$D_x = 1.337$ Mg m^{-3}
$M_r = 387.45$	Cu $K\alpha$ radiation
Monoclinic, $C2$	Cell parameters from 20
$a = 19.972$ (3) Å	reflections
$b = 6.509$ (3) Å	$\theta = 41.4$–$42.6°$
$c = 15.472$ (3) Å	$\mu = 0.78$ mm^{-1}
$\beta = 106.874$ (13)°	$T = 122$ (2) K
$V = 1924.6$ (10) Å^3	Rod, colourless
$Z = 4$	$0.35 \times 0.10 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 74.9°$
	$h = -24 \rightarrow 23$
ω–2θ scans	$k = -8 \rightarrow 8$
Absorption correction: none	$l = -19 \rightarrow 19$
3940 measured reflections	5 standard reflections
3940 independent reflections	frequency: 167 min
3841 reflections with $I > 2\sigma(I)$	intensity decay: none

organic papers

Refinement

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—HO3$\cdots$O2	0.80 (3)	2.14 (2)	2.638 (1)	120 (2)
O18—HO18$\cdots$O$W1^i$	0.94 (2)	1.78 (2)	2.709 (1)	171 (2)
N2—HN2$\cdots$O1^{ii}	1.03 (2)	1.66 (2)	2.685 (1)	175 (2)
O$W1$—H$W1A\cdots$O1^{iii}	0.84 (3)	1.87 (3)	2.692 (2)	166 (2)
O$W1$—H$W1B\cdots$O2^{iv}	0.89 (4)	2.06 (4)	2.931 (2)	167 (4)

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

The determination of the absolute configuration is based on the known configuration of the (R,R)-tartaric acid used in the preparation, but the refined Flack (1983) parameter, while having a relatively large uncertainty, is in accord with it. All the H atoms were clearly visible in the difference electron-density map and were fixed in idealized positions with an isotropic displacement parameter of 1.5 times that of the parent atom.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *DREADD* (Blessing, 1989); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *PLATON*94 (Spek, 1994).

References

Blessing, R. H. (1989). *J. Appl. Chem.* **22**, 396–397.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Gjerløv, A. & Larsen, S. (1997). *Acta Cryst.* B**53**, 708–718.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
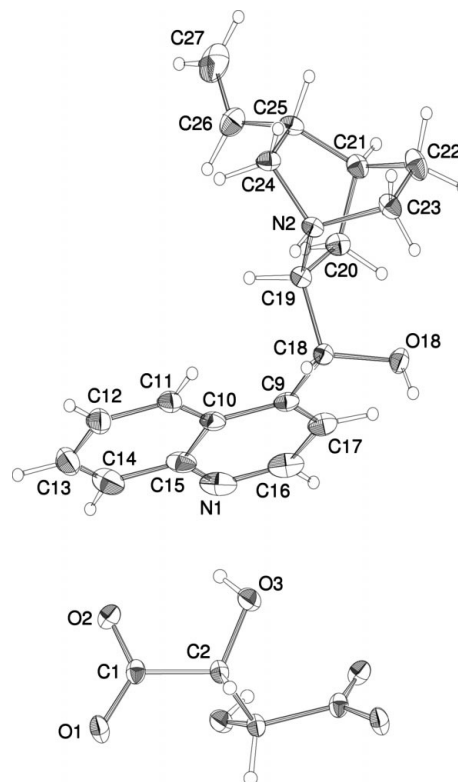Ryttersgaard, C. & Larsen, S. (1998). *Acta Cryst.* C**54**, 1698–1701.

Figure 1
An *ORTEP*II (Johnson, 1976) drawing of the ion pair. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres with a fixed radius.
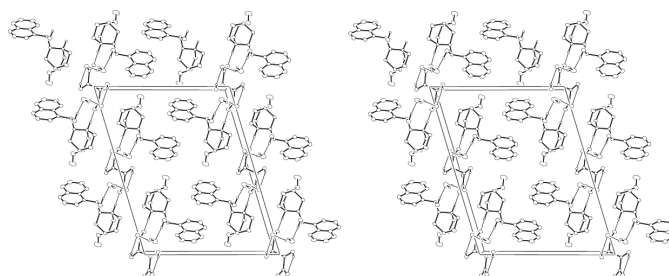

Figure 2
A stereopair illustrating the crystal packing. The structure is viewed along the *b* axis, with the *c* axis horizontal. The intermolecular hydrogen bonds are drawn as thin lines.

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