Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis(quininium) (*R*,*R*)-tartrate monohydrate

Carsten Ryttersgaard* and Sine Larsen

Centre for Crystallographic Studies, Department of Chemistry, Universitetsparken 5, DK-2100 Copenhagen, Denmark

Correspondence e-mail: carsten@ccs.ki.ku.dk

Received 19 September 2003
Accepted 21 November 2003
Online 6 December 2003

A disordered tartrate ion of the title compound, $2C_{20}H_{25}N_2O_2\cdot C_4H_4O_6\cdot H_2O$, is located on the crystallographic twofold axis. Hydrogen bonds are formed between the carboxylate group and the quinuclidine group of the protonated quinine ion.

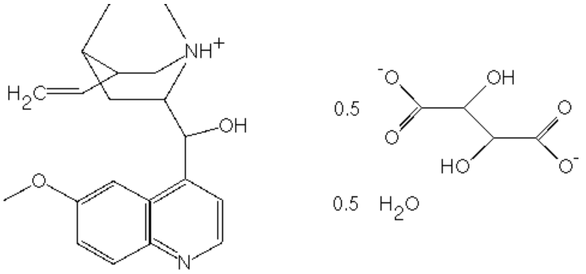
Key indicators

Single-crystal X-ray study
T = 122 K
Mean σ(C–C) = 0.005 Å
H-atom completeness 90%
Disorder in solvent or counterion
R factor = 0.057
wR factor = 0.154
Data-to-parameter ratio = 10.0

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

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 bis(18*R*,19*S*,21*S*,25*S*)-quininium (2*R*,3*R*)-tartrate monohydrate. Quinine differs from cinchonidine only by having an additional methoxy group attached to the quinoline ring system. Though the crystal packing of the title salt is similar to the packing in cinchonidinium (2*R*,3*R*)-tartrate (Ryttersgaard & Larsen, 2003), there are some distinct differences between the two crystal structures. Their packing arrangements resemble those found in mandelate salts of Cinchona alkaloids (Gjerløv & Larsen, 1997) and not in the hydrogen tartrate of quinine (Ryttersgaard & Larsen, 1998), but whereas the cinchonidinium tartrate is an ordered structure, the quininium tartrate crystallizes as a hydrate and displays significant disorder. The disorder can be described as two different populations of the anion and water molecule.



(I)

Experimental

Crystals were obtained by mixing equimolar amounts of 96% ethanol solutions of quinine and (*R*,*R*)-tartaric acid.

Crystal data

$2C_{20}H_{25}N_2O_2^+\cdot C_4H_4O_6^{2-}\cdot H_2O$	D_x = 1.351 Mg m^{-3}
M_r = 816.94	Mo $K\alpha$ radiation
Monoclinic, $C2$	Cell parameters from 20 reflections
a = 20.352 (3) Å	θ = 17.8–20.9°
b = 6.6070 (16) Å	μ = 0.10 mm^{-1}
c = 15.501 (2) Å	T = 122 (2) K
β = 105.580 (12)°	Rod, colourless
V = 2007.8 (6) Å^3	0.33 × 0.15 × 0.10 mm
Z = 2	

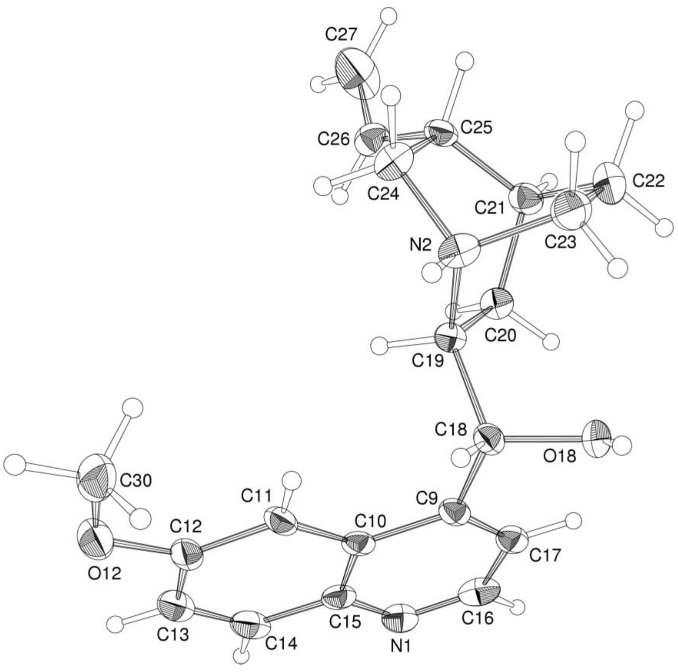


Figure 1
*ORTEP*III (Burnett & Johnson, 1996) drawing of the cation in the title compound. Displacement ellipsoids are drawn at the 50% probability level, and the H atoms are drawn as spheres with a fixed radius.

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
3165 measured reflections
3165 independent reflections
2533 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 30.0°$
$h = -28 \rightarrow 27$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 21$
5 standard reflections
frequency: 167 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.154$
$S = 1.09$
3165 reflections
318 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0783P)^2 + 1.2182P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.53$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$\cdots$O6	0.90	1.92	2.790 (7)	163
N2—H2$\cdots$O2$^{\rm i}$	0.90	1.78	2.662 (9)	167
O18—H180$\cdots$O70$^{\rm ii}$	0.84	1.78	2.618 (7)	174
O18—H180$\cdots$O1$^{\rm i}$	0.84	2.32	3.053 (8)	143

Symmetry codes: (i) $-x, y, -z$; (ii) $x, 1+y, z$.

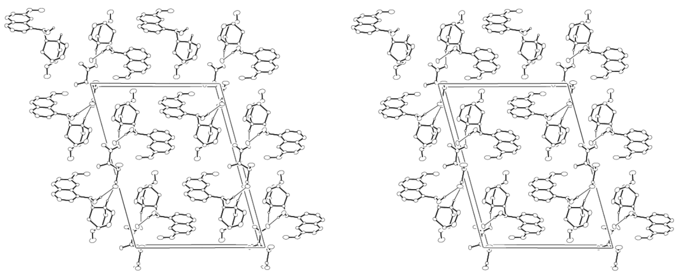


Figure 2
A stereopair illustrating the crystal packing. The structure is viewed along the *b* axis with the *c* axis horizontal. The intermolecular hydrogen bonds are drawn as thin lines.

The tartrate ion is located on a crystallographic twofold axis, where it adopts two different conformations each refined with 50% occupancy. In one of the conformations, the tartrate ion forms a hydrogen bond to a water molecule that thus also is half populated. H atoms of the tartrate ion and water molecule could not be identified in the electron density, whereas it was possible to identify the H atoms in the cation. The crystal packing is influenced by hydrogen bonds between the cation and the anion in its two conformations. The disorder of the anion over a crystallographic symmetry element leads to bad contacts between the atoms of the anion and the water molecule. The determination of the absolute configuration is based on the known configuration of the (*R*,*R*)-tartaric acid used in the preparation. Only the H atoms in quinine were visible in the difference electron-density map and these were fixed in idealized positions (sp^3 hybridized C—H = 0.99 Å, sp^2 hybridized C—H = 0.95 Å, N—H = 0.90 Å and O—H = 0.84 Å) with an isotropic displacement parameter of $1.5U_{\rm eq}$ of the parent atom, and treated as riding.

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*90 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *PLATON* (Spek, 2003).

We thank Mr Flemming Hansen for technical assistance with the data collection. This work was supported by a grant from The Danish Natural Science Research Council.

References

Blessing, R. H. (1989). *J. Appl. Chem.* **22**, 396–397.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Enraf–Nonius (1989). *CAD*-4 *Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Gjerløv, A. & Larsen, S. (1997). *Acta Cryst.* B**53**, 708–718.
Ryttersgaard, C. & Larsen, S. (1998). *Acta Cryst.* C**54**, 1698–1701.
Ryttersgaard, C. & Larsen, S. (2003). *Acta Cryst.* E**59**, o1715–o1716.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.