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1-Carboxylatomethyl-2-(hydroxyquinolin-4-yl methyl)-5-vinyl-1-azoniabicyclo[2.2.2]octane, a zwitterion of the Cinchona alkaloid complex

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The title compound, $C_{21}H_{24}N_2O_3$, is a zwitterion of the Cinchona alkaloid complex. In the crystal structure, intermolecular O—H···O and C—H···O hydrogen bonds link the molecules into one-dimensional double-stranded ladders. The crystal packing is further stabilized by van der Waals forces.

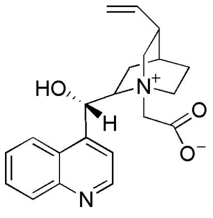
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
R factor = 0.060
wR factor = 0.117
Data-to-parameter ratio = 9.1

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

Comment

Cinchona alkaloids are well known in medicine and chemistry (Kacprzak & Gawronski, 2001; Taggi *et al.*, 2003). Cinchona alkaloid-derived quaternary ammonium salts are exploited in the chiral phase-transfer catalyst (PTC) process (O'Donnell, 2000, 2001; Lygo, 2001; Shen *et al.*, 2003) as inexpensive and attractive organocatalysts. In the study of the mechanism of the reaction, knowledge of preferred conformations of catalysts and some knowledge of non-covalent interactions is of crucial importance. Conformational analyses of Cinchona alkaloids and their derivatives in solution and in the solid state have been carried out before by Wynberg and others (Wynberg *et al.*, 1989; Dijkstra *et al.*, 1990; Aune *et al.*, 1995). Cannizzaro & Houk (2002) demonstrated that Cinchona alkaloid-derived PTCs possess special hydrogen bonds of the type $N^+—CH_2···O=C$, which play an important role in asymmetric PTC reactions. In our study we obtained the title compound, (I) (Fig. 1) and report its crystal structure here.



(I)

The quinoline group of (I) is planar to within 0.02 Å. The torsion angles C15—C16—C9—O1 (−14.7°), O1—C12—C1—C2 (61.5°) and O1—C12—C1—N1 (−62.6°), which determine the conformation of (I), are significantly different from those in cinchonine base (Oleksyn & Ciechanowicz-Rutkowska, 1979). The quinuclidine group has a very twisted conformation which is far from the D_{3h} symmetry of the ideal bicyclooctane nucleus.

In the crystal structure, intermolecular O—H···O and C—H···O hydrogen bonds (Table 1) link the molecules into one-dimensional double-stranded ladders. The C—H···O hydrogen bonds in (I) are a special type of $N^+—CH_2···O=C$ interactions reported by Cannizzaro & Houk (2002). The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

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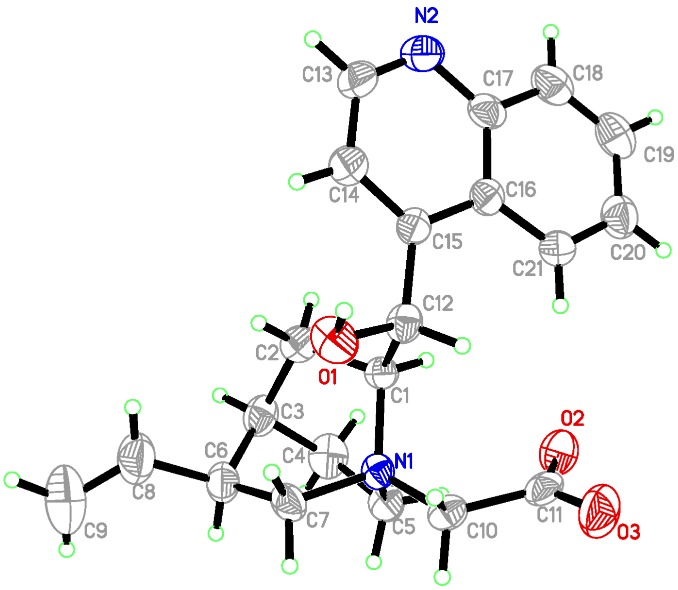

Figure 1
View of (I), showing 30% probability displacement ellipsoids and the atom-labelling scheme.

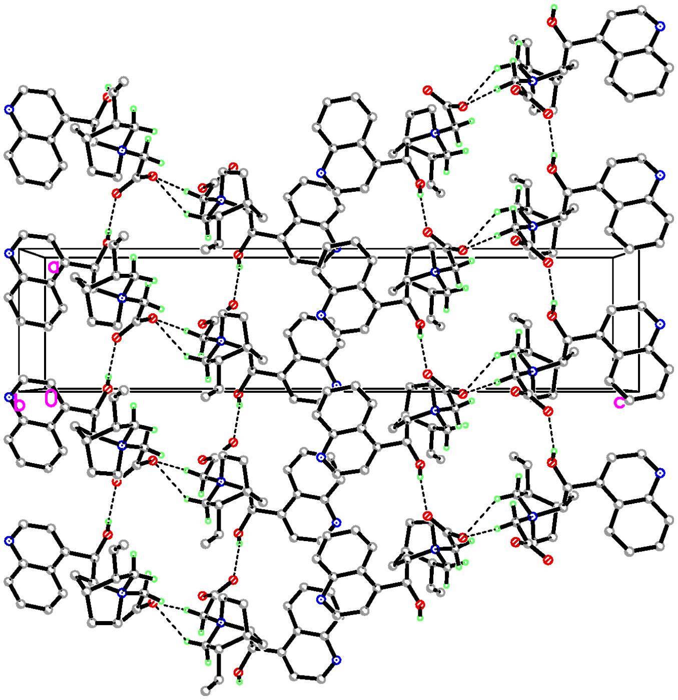

Figure 2
Packing diagram and the hydrogen-bonding (dashed lines) network, viewed along the *b* axis.

Experimental

Compound (I) was prepared by the reaction of cinchonine iodoacetamide with a solution of silver chloride in water, in accordance with the literature (Walter & Heidelberger, 1919). Single crystals suitable for X-ray diffraction study were obtained from methanol by slow evaporation at room temperature.

Crystal data

$C_{21}H_{24}N_2O_3$
$M_r = 352.42$
Orthorhombic, $P2_12_12_1$
$a = 6.952$ (2) Å
$b = 8.522$ (3) Å
$c = 30.120$ (9) Å
$V = 1784.4$ (9) Å^3
$Z = 4$
$D_x = 1.312$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 869 reflections
$\theta = 2.5$–$24.0°$
$\mu = 0.09$ mm^{-1}
$T = 292$ K
Block, colourless
0.22 × 0.18 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.981$, $T_{\max} = 0.989$
10346 measured reflections

2144 independent reflections
1694 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 26.4°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -17 \rightarrow 37$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.117$
$S = 1.08$
2144 reflections
236 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.0926P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$O2$^{\rm i}$	0.82	1.99	2.712 (3)	147
C7—H7$B\cdots$O3$^{\rm ii}$	0.97	2.52	3.353 (5)	144
C10—H10$A\cdots$O3$^{\rm ii}$	0.97	2.40	3.291 (4)	152

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms and 1.2$U_{\rm eq}$(C) for other H atoms. Each methyl group was allowed to rotate freely about its C—C bond. Due to the absence of any significant anomalous scatterers in the molecule, attempts to confirm the absolute configuration by refinement of the Flack (1983) parameter in the presence of 1527 sets of Friedel equivalents led to an inconclusive value of −0.4 (19). Therefore, the Friedel pairs were merged before the final refinement and the absolute configuration was assigned by reference to an unchanging chiral centre in the synthetic procedure.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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