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Key indicators

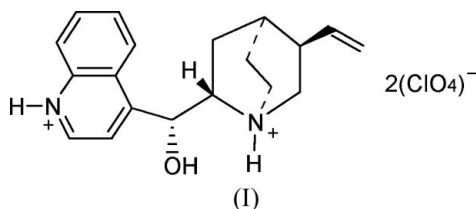
Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.043
 wR factor = 0.121
Data-to-parameter ratio = 14.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Cinchonidinium bis(perchlorate)

In the title crystal structure, $\text{C}_{19}\text{H}_{24}\text{N}_2\text{O}^{2+} \cdot 2\text{ClO}_4^-$, cations and anions are connected by intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds, forming one-dimensional chains propagating in the a -axis direction. These chains are, in turn, linked by weak $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds, forming a three-dimensional network.

Comment

The crystal structures of some cinchonidine compounds have already been reported, *e.g.* cinchonidinium (*S*)-mandelate (Gjerlov & Larsen, 1997), bis(cinchonidinium) *L*-tartrate dihydrate (Zhang *et al.*, 2003), cinchonidinium (*R,R*)-tartrate monohydrate (Ryttersgaard & Larsen, 2003) and cinchonidinium trichlorocobalt(II) (Skorska *et al.*, 2005). The molecular structure of the title compound, (I), is shown in Fig. 1.



One anion of (I) is connected to the cation through an $\text{O}-\text{H} \cdots \text{O}$ hydrogen bond and the other through an $\text{N}-\text{H} \cdots \text{O}$ hydrogen bond (Table 2). Within the cation, two weak $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds may influence the molecular confor-

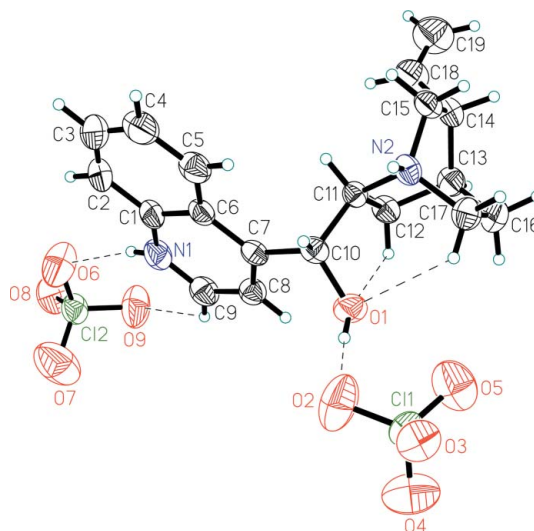
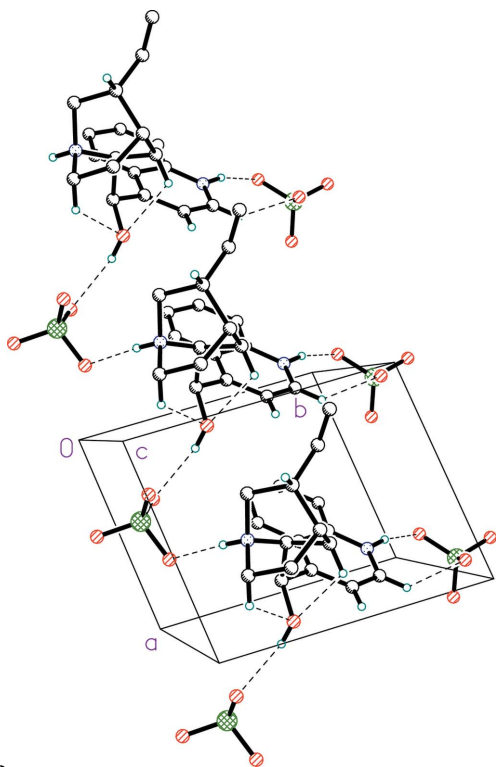


Figure 1

The asymmetric unit of (I), showing 40% probability displacement ellipsoids. Dashed lines denote hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

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**Figure 2**

A hydrogen-bonded chain of (I) along the [100] direction. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

mation. In the crystal structure, anions and cations are linked by intermolecular N—H···O and O—H···O hydrogen bonds, forming one-dimensional chains running along the *a* axis (Fig. 2). These chains are, in turn, connected by weak intermolecular C—H···O hydrogen bonds, forming a three-dimensional network (Fig. 3).

Experimental

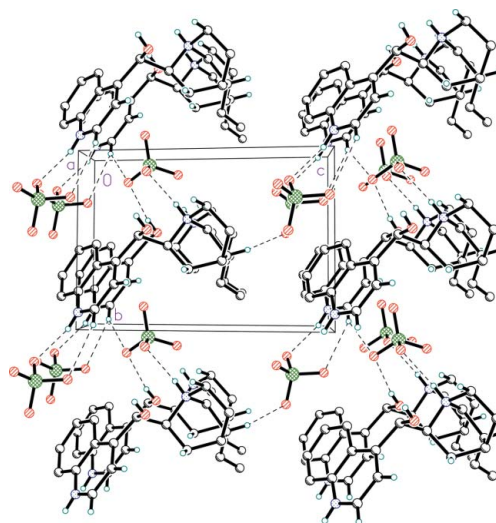
Cinchonidine (1 mmol, 0.29 g) and 10% aqueous perchloric acid (2 mmol, 0.20 g) were mixed and water (10 ml) added, then heated to 373 K and stirred for half an hour. The reaction system was cooled to room temperature and colorless crystals were collected after 10 d.

Crystal data

$C_{19}H_{24}N_2O^{2+} \cdot 2ClO_4^-$	$V = 560.6 (2) \text{ \AA}^3$
$M_r = 495.30$	$Z = 1$
Triclinic, <i>P1</i>	$D_x = 1.467 \text{ Mg m}^{-3}$
$a = 6.544 (1) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.819 (2) \text{ \AA}$	$\mu = 0.34 \text{ mm}^{-1}$
$c = 11.308 (2) \text{ \AA}$	$T = 292 (2) \text{ K}$
$\alpha = 88.67 (2)^\circ$	Block, colorless
$\beta = 77.03 (1)^\circ$	$0.48 \times 0.42 \times 0.42 \text{ mm}$
$\gamma = 83.85 (2)^\circ$	

Data collection

Siemens P4 diffractometer	4392 independent reflections
ω scans	3864 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$\theta_{\max} = 26.0^\circ$
$T_{\min} = 0.850$, $T_{\max} = 0.869$	3 standard reflections
4392 measured reflections	every 97 reflections
	intensity decay: 4.1%

**Figure 3**

Part of the crystal structure of (I), with hydrogen bonds shown as dashed lines.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0806P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.121$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.06$	$\Delta\rho_{\max} = 0.32 \text{ e \AA}^{-3}$
4390 reflections	$\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$
302 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.059 (7)
	Absolute structure: Flack (1983), 2195 Friedel pairs
	Flack parameter: $-0.01 (6)$

Table 1

Selected geometric parameters (\AA , $^\circ$).

C11—O2	1.416 (3)	N2—C17	1.497 (4)
C12—O9	1.437 (3)	C8—C9	1.363 (5)
C12—O6	1.443 (3)	C10—C11	1.546 (4)
O1—C10	1.422 (4)	C14—C18	1.514 (5)
N1—C9	1.317 (5)	C18—C19	1.269 (6)
O3—C11—O2	111.0 (2)	C2—C1—C6	121.9 (3)
O7—C12—O9	110.3 (3)	N1—C9—C8	120.3 (3)
O7—C12—O6	113.1 (3)	O1—C10—C11	108.9 (2)
O9—C12—O6	106.7 (2)	N2—C11—C10	112.8 (2)
C9—N1—C1	123.5 (3)	N2—C15—C14	109.8 (2)
C17—N2—C15	109.3 (2)	C19—C18—C14	124.2 (4)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1O···O2	0.82 (4)	2.08 (4)	2.889 (5)	167 (4)
N1—H1N···O6	0.85 (3)	2.04 (3)	2.867 (4)	163 (3)
N2—H2N···O4 ⁱ	0.86 (3)	2.05 (3)	2.900 (4)	175 (3)
C8—H8···O1	0.93	2.42	2.760 (6)	102
C9—H9···O2 ⁱⁱ	0.93	2.57	3.448 (5)	158
C9—H9···O9	0.93	2.58	3.192 (5)	124
C12—H12B···O1	0.97	2.39	2.856 (4)	109
C14—H14···O8 ⁱⁱⁱ	0.98	2.54	3.402 (3)	147
C17—H17A···O1	0.97	2.48	3.116 (5)	123

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

H atoms bonded to O and N atoms were located in difference Fourier maps and refined isotropically with O—H and N—H distances restrained to 0.82 (1) and 0.86 (1) Å, respectively. All other H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.93 (aromatic and alkene), 0.97 (methyl) and 0.98 Å (methine), with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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