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Cinchonidinium bis(4-methylbenzenesulfonate) monohydrate

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.005 Å
R factor = 0.042
wR factor = 0.113
Data-to-parameter ratio = 14.6

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

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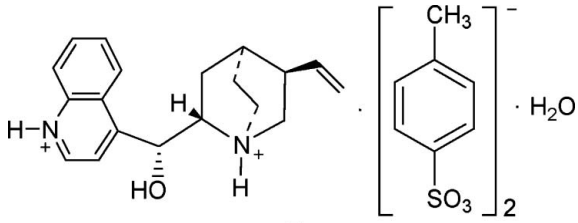
In the title salt, $C_{19}H_{24}N_2O^{2+}\cdot 2C_7H_7SO_3^-\cdot H_2O$, the ions and the water molecule are held together by O—H···O, N—H···O and C—H···O hydrogen bonds, forming hydrogen-bonded layers; there are no hydrogen bonds between layers.

Comment

Various salts of cinchonidine have been reported previously, *e.g.* cinchonidinium pyruvate oxime (Borszeky *et al.*, 1996), cinchonidinium (*S*)-mandelate (Gjerløv & Larsen, 1997), bis(cinchonidinium) L-tartrate dihydrate (Zhang *et al.*, 2003), cinchonidinium (*R*,*R*)-tartrate monohydrate (Ryttersgaard & Larsen, 2003), cinchonidinium tris(tetrachlorobenzenediolato)phosphate (Favarger *et al.*, 2004) and cinchonidinium bis(perchlorate) (Zhang *et al.*, 2006). As a continuation of our research, we report here the title salt, (I).



(I)

As shown in Fig. 1, the 4-methylbenzenesulfonate anion containing atom S1, the cinchonidinium cation and the water molecule are held together by N—H···O, O—H···O and C—H···O hydrogen bonds (Table 2) in an $R_3^3(9)$ ring (Etter, 1990).

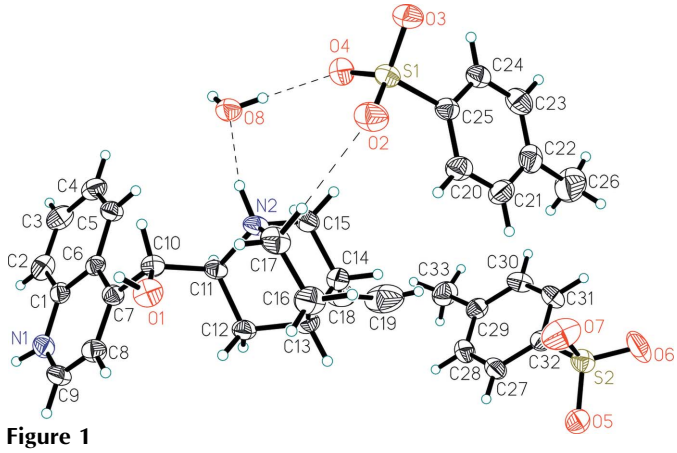


Figure 1
The asymmetric unit of (I), showing the atom labelling and 40% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

Further N—H···O, O—H···O and some weak C—H···O hydrogen bonds (Table 2) form a layer parallel to the (001) plane (Fig. 2). The whole structure is generated though translation of the layer; there are no hydrogen bonds between layers.

Experimental

Cinchonidine and 4-methylbenzenesulfonic acid in a 1:3 molar ratio were mixed and dissolved with stirring in sufficient water by heating to 373 K, at which point a clear solution resulted. The solution was cooled to room temperature and the colourless crystals were collected after 30 d.

Crystal data

$C_{19}H_{24}N_2O^{2+}\cdot 2C_7H_7SO_3^-\cdot H_2O$
M_r = 656.81
Triclinic, $P\overline{1}$
a = 6.565 (3) Å
b = 9.599 (4) Å
c = 12.748 (5) Å
α = 85.25 (3)°
β = 84.08 (3)°
γ = 82.71 (3)°
V = 790.6 (6) Å^3
Z = 1
D_x = 1.380 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 291 (2) K
Block, colourless
0.54 × 0.42 × 0.26 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: ψ scan (*XSCANS*; Siemens, 1994)
T_{min} = 0.932, T_{max} = 0.994
(expected range = 0.884–0.943)
6402 measured reflections
6198 independent reflections
5316 reflections with $I > 2\sigma(I)$
R_{int} = 0.000
θ_{max} = 26.0°
3 standard reflections
every 97 reflections
intensity decay: 4.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
S = 1.05
6198 reflections
425 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 0.0459P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.016 (3)
Absolute structure: Flack (1983), 2176 Friedel pairs
Flack parameter: −0.03 (6)

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.438 (3)	C1—C2	1.395 (4)
S1—O3	1.439 (2)	C5—C6	1.413 (4)
S1—O4	1.460 (3)	C10—C11	1.540 (4)
S1—C25	1.775 (3)	C14—C18	1.502 (5)
S2—O5	1.462 (2)	C18—C19	1.293 (6)
O1—C10	1.413 (4)	C20—C25	1.382 (4)
N2—C11	1.509 (4)	C21—C22	1.379 (6)
O2—S1—O3	114.45 (15)	O1—C10—C7	111.1 (2)
O2—S1—C25	106.39 (15)	C7—C10—C11	109.0 (2)
O7—S2—O6	116.4 (2)	C13—C12—C11	109.0 (2)
C9—N1—C1	123.2 (3)	C19—C18—C14	124.7 (5)
C15—N2—C11	107.6 (2)	C21—C22—C23	117.8 (3)
C2—C3—C4	120.5 (3)	C24—C25—C20	119.5 (3)

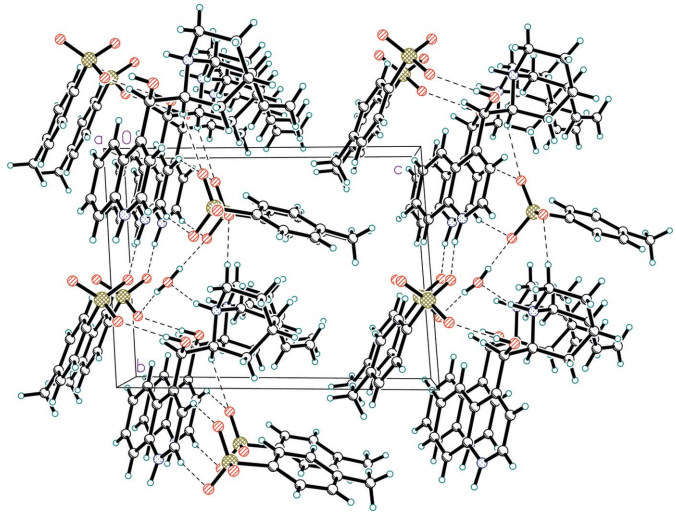


Figure 2
A packing diagram of (I), viewed along the *a* axis. Hydrogen bonds are indicated by dashed lines.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O6^{i}	0.98 (6)	1.86 (6)	2.749 (4)	150 (5)
N2—H2N···O8	0.99 (4)	1.75 (4)	2.718 (3)	164 (4)
O1—H1O···O5^{ii}	0.82	1.96	2.754 (3)	163
O8—H0*A*···O4	0.82 (1)	1.91 (1)	2.731 (4)	179 (5)
O8—H0*B*···O5^{iii}	0.83 (1)	1.98 (1)	2.806 (3)	172 (4)
C8—H8···O3^{iv}	0.93	2.46	3.205 (4)	138
C9—H9···O4^{iv}	0.93	2.34	3.247 (4)	164
C11—H11···O3^{v}	0.98	2.51	3.320 (4)	140
C17—H17*A*···O1	0.97	2.39	3.026 (4)	123
C17—H17*B*···O2	0.97	2.50	3.441 (3)	164

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

H atoms of the water molecule and those attached to N atoms were located in difference Fourier maps. Water H atoms were refined with O—H and H···H distances restrained to 0.82 (1) and 1.34 (1) Å, respectively, while the H atoms of the NH group were refined freely. 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), 0.96 (methyl), 0.97 (methylene), 0.98 (methine) and 0.82 Å (hydroxyl), with U_{iso}(H) = 1.2U_{eq}(C,O).

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*.

References

Borszeky, K., Mallat, T., Aeschiman, R., Schweizer, W. B. & Baiker, A. (1996). *J. Catal.* **161**, 451–458.
Bruker (1998). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Favarger, F., Goujion-Ginglinger, C., Monchand, D. & Lacour, J. (2004). *J. Org. Chem.* **69**, 8521–8524.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Gjerløv, A. & Larsen, S. (1997). *Acta Cryst.* B**53**, 708–718.
Ryttersgaard, C. & Larsen, S. (2003). *Acta Cryst.* E**59**, o1715–o1716.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1994). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Zhang, H., Lin, Z.-Y., Zhou, Z.-H. & Ng, S. W. (2003). *Acta Cryst.* E**59**, o183–o185.
Zhang, H.-M., Tu, B., Jin, Z.-M., Wu, Z.-Y. & Hu, M.-L. (2006). *Acta Cryst.* E**62**, o2614–o2616.