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Cinchoninium hydrogen isophthalate at 153 K

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.124
Data-to-parameter ratio = 9.6

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

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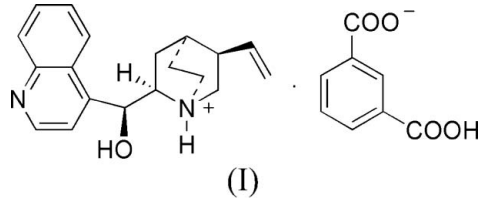
In the title compound, $C_{19}H_{23}N_2O^+\cdot C_8H_5O_4^-$, the cation and anion are held together by an N—H···O hydrogen bond. One-dimensional chains along the [100] direction are formed *via* O—H···O hydrogen bonds. The chains associate by O—H···N and C—H···O hydrogen bonds to form the complete structure.

Comment

There are various diastereoisomers, such as cinchonidine and cinchonine, in cinchona alkaloids. Supramolecular crystallization is a unique approach for separating isomers. Some superamolecular compounds containing cinchonine have been reported previously (Oleksyn *et al.*, 1978; Larsen *et al.*, 1993; Dyrek *et al.*, 1997; Puliti *et al.*, 2001). Recently, the title compound, (I), was synthesized in our laboratory.



(I)

Compound (I), is composed of a cinchoninium cation and a hydrogen isophthalate anion (Fig. 1), which are linked by

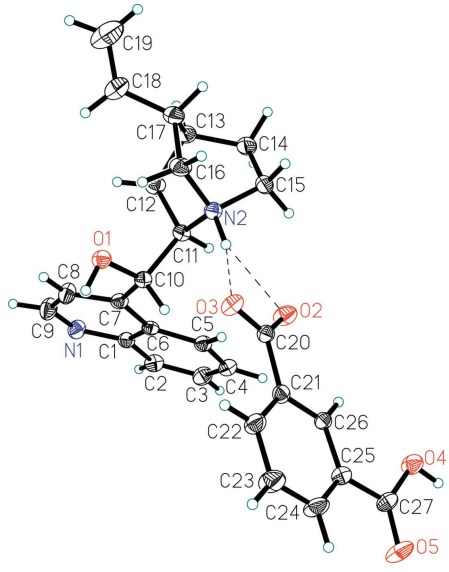


Figure 1
The asymmetric unit of (I), with the atom-labelling scheme, showing 40% probability displacement ellipsoids. The thin dashed lines denote the hydrogen bonds.

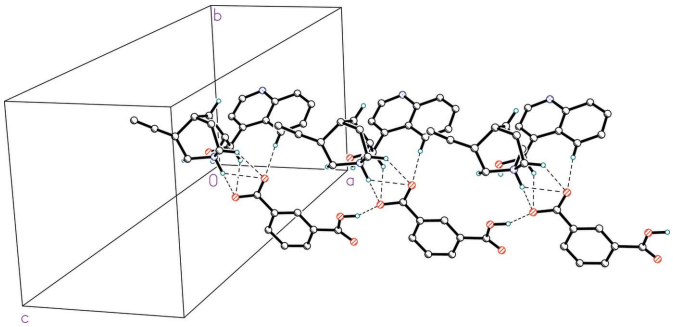


Figure 2
The hydrogen-bonded chains of (I) along the [100] direction. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

N—H···O and C—H···O hydrogen bonds. (Table 2). The geometry of the cinchoninium cation (Table 1) is consistent with our previous results (Larsen *et al.*, 1993; Puliti *et al.*, 2001). In the cinchoninium cation, the C16—H16*B*···O1 hydrogen bond may influence the molecular conformation.

The carboxyl group and the carboxylate of a neighbouring anion are connected by the O4—H4*O*···O3^{i} hydrogen bond to form one-dimensional chains along the *a* axis (Fig. 2 and Table 2). Adjacent hydrogen bonded chains associate by the O1—H1*O*···N1^{ii} and C12—H12*B*···O5^{iii} hydrogen bonds to complete the supramolecular three-dimensional structure (Table 2 and Fig. 3).

Experimental

Cinchonine (Aldrich, 1 mmol, 0.29 g) and isophthalic acid (1 mmol, 0.17 g) were dissolved in 10 ml ethanol and 10 ml water, then heated to boiling and stirred for ten minutes. The reaction system was cooled to room temperature and colourless crystals were collected after four days.

Crystal data

$C_{19}H_{23}N_2O^+\cdot C_8H_5O_4^-$
M_r = 460.51
Orthorhombic, $P2_12_12_1$
a = 8.5208 (6) Å
b = 10.2582 (8) Å
c = 26.8066 (18) Å
V = 2343.1 (3) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 153 (2) K
Block, colourless
0.44 × 0.23 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
22585 measured reflections
3056 independent reflections
2531 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.124
S = 1.01
3056 reflections
320 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0866P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0077 (15)

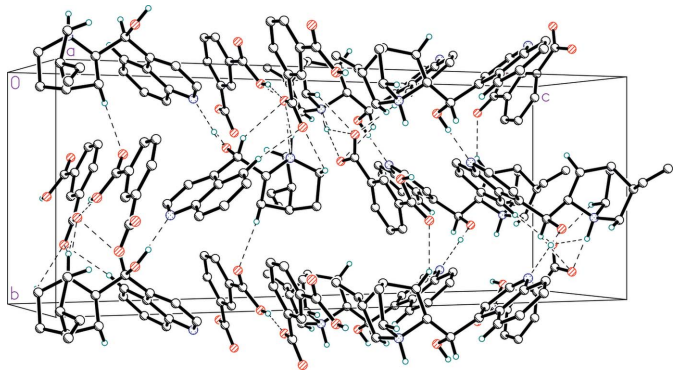


Figure 3
The packing of (I), with hydrogen bonds shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Table 1
Selected geometric parameters (Å, °).

O1—C10	1.408 (3)	C10—C11	1.530 (3)
O2—C20	1.237 (3)	C13—C14	1.531 (4)
O3—C20	1.274 (3)	C17—C18	1.506 (4)
O4—C27	1.321 (4)	C18—C19	1.296 (5)
O5—C27	1.210 (3)	C20—C21	1.506 (4)
N1—C1	1.371 (3)	C21—C22	1.389 (4)
N2—C15	1.500 (3)	C25—C27	1.489 (3)
C1—C2	1.410 (3)		
C15—N2—C16	108.49 (19)	C19—C18—C17	124.8 (4)
N1—C1—C2	117.4 (2)	O2—C20—O3	123.5 (2)
C3—C2—C1	120.3 (2)	O2—C20—C21	118.7 (2)
O1—C10—C7	111.86 (19)	C22—C21—C26	119.3 (2)
C7—C10—C11	108.9 (2)	C22—C21—C20	122.8 (2)
N2—C11—C12	107.86 (19)	C24—C25—C27	119.9 (3)
C12—C11—C10	115.5 (2)	O5—C27—O4	123.9 (3)
C11—C12—C13	108.1 (2)	O5—C27—C25	123.9 (3)
C18—C17—C13	113.1 (3)	O4—C27—C25	112.2 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*O*···O3^{i}	0.99 (5)	1.64 (5)	2.572 (3)	154 (5)
O1—H1*O*···N1^{ii}	0.92 (4)	1.80 (4)	2.713 (3)	171 (4)
N2—H2*N*···O2	1.06 (4)	2.39 (4)	3.074 (5)	121 (3)
N2—H2*N*···O3	1.06 (4)	1.69 (4)	2.734 (3)	168 (3)
C5—H5···O2	0.95	2.48	3.419 (6)	171
C10—H10···O3	1.00	2.50	3.225 (3)	129
C12—H12*B*···O5^{iii}	0.99	2.48	3.158 (2)	126
C15—H15*A*···O2	0.99	2.58	3.224 (3)	123
C16—H16*B*···O1	0.99	2.29	2.978 (5)	126

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

H atoms bonded to N and O atoms were located in difference Fourier maps and refined freely. Other H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.95 (aromatic and alkene C—H), 0.99 (methylene), 1.00 Å (methine), with U_{iso}(H) = 1.2U_{eq}(C). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure:

SHELXL97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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