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Dicyclohexylaminium 4-hydroxybenzenesulfonate monohydrate

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In the title salt, $C_{12}H_{24}N^+\cdot C_6H_5O_4S^-\cdot H_2O$, the dicyclohexylaminium cation is linked to the 4-hydroxysulfonate anion and water molecules *via* N—H···O and O—H···O hydrogen bonds. The water molecule acts as both a hydrogen donor and acceptor in establishing a three-dimensional hydrogen-bonded network.

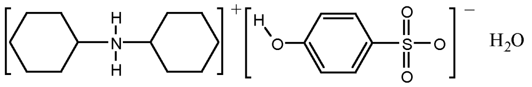
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.133
Data-to-parameter ratio = 14.5

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

Comment

Dicyclohexylamine is mainly used for synthesizing rubber accelerators and metal corrosion inhibitors. In the past, some supramolecular complexes were synthesized with dicyclohexylamine (Nakamura *et al.*, 1996; Yutronic *et al.*, 2001; Guo *et al.*, 2002; Baranowska *et al.*, 2003; Smith *et al.*, 2004). We report here the structure of the title salt, (I), which was obtained from 4-hydroxysulfonic acid, dicyclohexylamine and water.



(I)

Fig. 1 shows the molecular structure of (I). The two rings of the dicyclohexylaminium cation adopt chair conformations. The bond lengths of S1—O3 [1.4439 (17) Å], S1—O4 [1.4421 (18) Å] and S1—O2 [1.4526 (17) Å] are normal for a

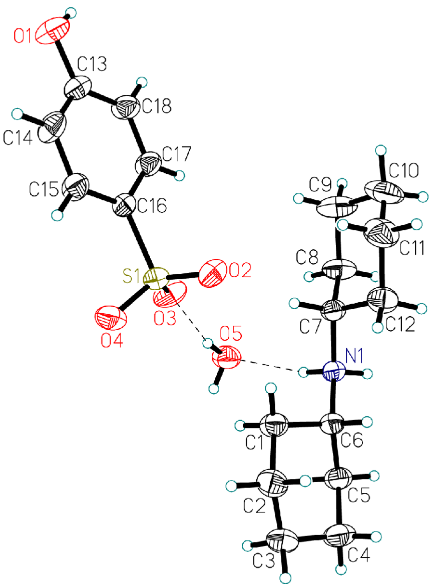


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

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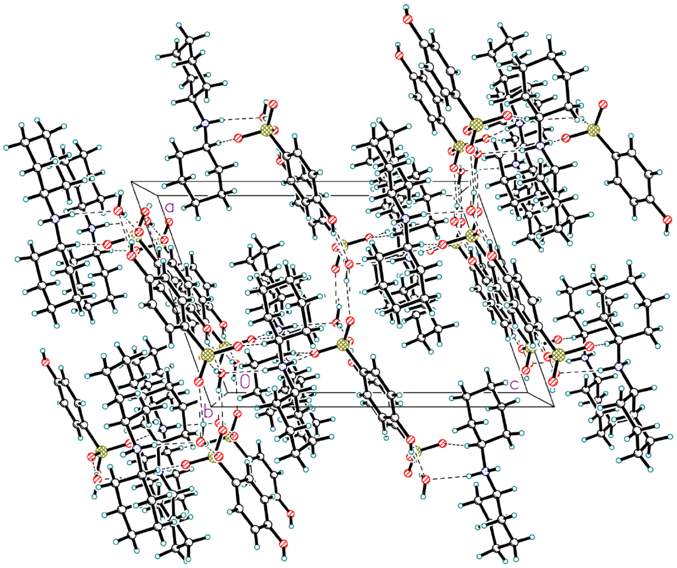

Figure 2
Packing of the molecules in (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

sulfonate group; the C—C bond lengths of the cyclohexane ring lie in the range 1.498 (5)–1.533 (3) Å (Table 1).

The dicyclohexylaminium cation is linked to the 4-hydroxysulfonate anion *via* an ionic $N1^{+}\cdots O2^{-i}$ interaction (see Table 2 for geometry details and symmetry code). The $N^{+}\cdots O^{-}$ distance [2.765 (3) Å] is shorter than the non-ionic N⋯O distance of 2.868 Å observed in an inclusion compound of 9,10-bis(1-naphthyl)phenanthrene-9,10-diol and dicyclohexylamine (Guo *et al.*, 2002).

Fig. 2 illustrates the unit-cell packing of (I). The water molecule acts as a hydrogen-bond donor for 4-hydroxysulfonate in O5—H⋯O3 and O5—H⋯$O4^{ii}$ hydrogen bonds, and as a hydrogen-bond acceptor in N1—H⋯O5 and O1—H⋯$O5^{iii}$ hydrogen bonds (see Table 2 for details). Two intramolecular C—H⋯O hydrogen bonds (Steiner & Desiraju, 1998), *viz.* C7—H7⋯O2 and C15—H15⋯O4 (Table 2), are observed in the title salt. The N—H⋯O and O—H⋯O hydrogen bonding in the structure leads to a three-dimensional hydrogen-bonded network.

Experimental

4-Hydroxysulfonic acid (0.01 mol), dicyclohexylamine (0.01 mol) and sufficient water were mixed and heated to form a clear solution. Crystals of the title salt were formed by gradual evaporation of water over a period of one week at 293 K.

Crystal data

$C_{12}H_{24}N^{+}\cdot C_6H_5O_4S^{-}\cdot H_2O$
$M_r = 373.50$
Monoclinic, $P2_1/n$
$a = 11.1091$ (4) Å
$b = 11.7800$ (3) Å
$c = 15.9282$ (5) Å
$\beta = 109.563$ (1)°
$V = 1964.12$ (11) Å^3
$Z = 4$
$D_x = 1.263$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3651 reflections
$\theta = 2.6$–45.0°
$\mu = 0.19$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.45 × 0.28 × 0.25 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.919$, $T_{max} = 0.954$
10211 measured reflections
3515 independent reflections
3168 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.2$°
$h = -9 \rightarrow 13$
$k = -12 \rightarrow 14$
$l = -19 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.134$
$S = 1.12$
3515 reflections
243 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.8528P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Selected bond lengths (Å).

N1—C6	1.498 (3)	C11—C12	1.533 (3)
N1—C7	1.505 (3)	S1—O4	1.4421 (18)
C1—C6	1.514 (3)	S1—O3	1.4439 (17)
C1—C2	1.527 (3)	S1—O2	1.4526 (17)
C2—C3	1.511 (4)	S1—C16	1.769 (2)
C3—C4	1.527 (4)	O1—C13	1.365 (3)
C4—C5	1.519 (3)	C13—C14	1.373 (3)
C5—C6	1.512 (3)	C13—C18	1.382 (3)
C7—C12	1.506 (3)	C14—C15	1.379 (3)
C7—C8	1.511 (3)	C15—C16	1.380 (3)
C8—C9	1.510 (4)	C16—C17	1.383 (3)
C9—C10	1.498 (5)	C17—C18	1.376 (3)
C10—C11	1.512 (4)		

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1*A*⋯O5	0.89 (2)	2.13 (2)	3.000 (3)	165 (2)
N1—H1*B*⋯$O2^{i}$	0.89 (3)	1.88 (3)	2.765 (3)	173 (2)
O5—H5*D*⋯O3	0.89 (4)	1.88 (4)	2.748 (3)	165 (3)
O5—H5*C*⋯$O4^{ii}$	0.83 (4)	1.97 (4)	2.778 (3)	165 (4)
O1—H1⋯$O5^{iii}$	0.82	2.05	2.839 (3)	162
C7—H7⋯O2	0.98	2.39	3.346 (3)	164
C15—H15⋯O4	0.93	2.58	2.928 (3)	103

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

H atoms attached to N1 and O5 were located in a difference map and were isotropically refined. All other H atoms were placed at calculated positions (O—H = 0.82 Å and C—H = 0.93–0.97 Å) and allowed to ride on their parent atoms, with $U_{iso}(H)$ set at $1.2U_{eq}(C)$ or $1.5U_{eq}(O)$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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