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Acta Crystallographica Section E
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Hydrogen bonding in *m*-carboxyphenylammonium bisulfate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.115
Data-to-parameter ratio = 19.4

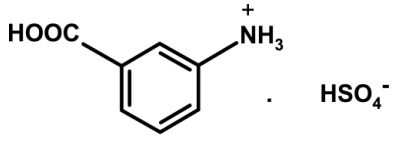
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 October 2003
Accepted 13 October 2003
Online 23 October 2003

In the title compound, $C_7H_8NO_2^+\cdot HSO_4^-$, there is an intricate cation–cation, cation–anion and anion–anion three-dimensional hydrogen-bond network. Cations are linked two-dimensionally by C—H···O hydrogen bonds, and anions are linked two-dimensionally by O—H···O hydrogen bonds; however, interactions between cations and anions involve a three-dimensional network of N—H···O and O—H···O hydrogen bonds. The strongest hydrogen bond is observed between anions.

Comment

Crystal engineering of organic–inorganic hybrid materials is currently of great interest and these materials have received increasing attention during the past few decades (Mazeaud *et al.*, 2000; Shogomonian *et al.*, 1998) owing to their interesting structural topologies and potential application in materials science, such as ion-exchange, adsorption, molecular recognition, catalysis and magnetism (Aakeroy *et al.*, 1999; Hagrman *et al.*, 1999). In our systematic investigation of organic–inorganic hybrid materials, including aminobenzoic acid and various inorganic acids, three structures have been already reported: *m*-carboxyphenylammonium nitrate (Benali-Cherif, Cherouana *et al.*, 2002), *p*-carboxyphenylammonium dihydrogenmonophosphate monohydrate (Benali-Cherif, Abouimrane *et al.*, 2002) and *m*-carboxyphenylammonium perchlorate (Bendjeddou *et al.*, 2003). In this paper, we describe our fourth crystal structure with aminobenzoic acid and a bisulfate ion, *m*-carboxyphenylammonium bisulfate, (I).



(I)

The carboxy moiety of the *m*-carboxyphenylammonium cation makes an angle of 11.4 (2)° with the benzene ring. The weighted average of the ring C—C bond lengths, 1.387 Å, is about 0.006 Å shorter than the value found in the crystalline form of benzene (Cox *et al.*, 1958).

The anion in compound (I) is monoprotonated and adopts a tetrahedral geometry; the bond distance between S and O5 confirms the presence of the H atom in the bisulfate [HSO_4^-] (Fig. 1). The crystal structure of (I) is built up from intricate cation–cation, anion–anion and anion–cation hydrogen-bond interactions in a three-dimensional network: (i) cation–cation

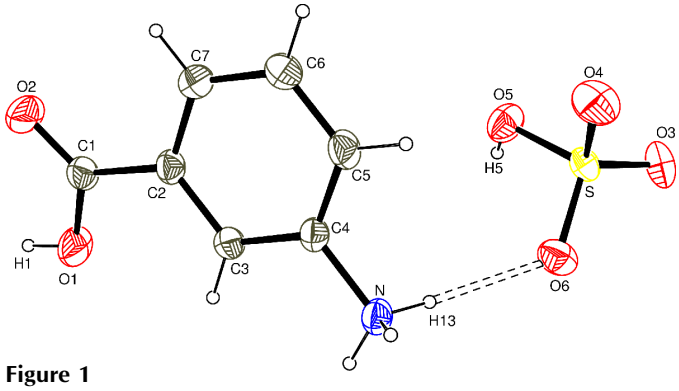


Figure 1
*ORTEP*III (Burnett & Johnson, 1996) view of the title compound, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

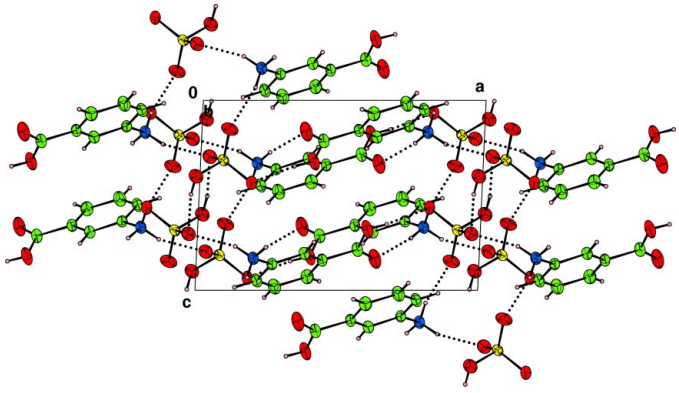


Figure 2
Packing view (*CAMERON*; Watkin *et al.*, 1993) of the intricate three-dimensional hydrogen-bond network forming a supramolecular structure.

interactions through N1—H11···O2^{i} form zigzag chains, which extend along the *b* axis; (ii) anion–anion interactions through O5—H5···O6^{iii} lead to the formation of dimers; and (iii) anion–cation interactions through N—H13···O6, O1—H1···O3^{iv} and N—H12···O4^{ii} assure the cohesion of the crystal trough the formation of a three-dimensional hydrogen-bond network. All these hydrogen-bonds interactions build a supramolecular structure, as shown in Fig. 2; geometrical details of hydrogen bonds and symmetry codes are given in Table 2.

Experimental

Brown single crystals of *m*-carboxyphenylammonium bisulfate were obtained after a few days by slow evaporation, at room temperature, of an equimolar aqueous solution of *m*-aminobenzoic and sulfuric acid.

Crystal data

$C_7H_8NO_2^+\cdot HSO_4^-$
M_r = 235.21
Monoclinic, $P2_1/c$
a = 11.4160 (3) Å
b = 10.5090 (3) Å
c = 7.6810 (2) Å
β = 92.562 (2)°
V = 920.57 (4) Å^3
Z = 4
D_x = 1.697 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12041 reflections
θ = 1.8–30.0°
μ = 0.36 mm^{-1}
T = 293 K
Prism, brown
0.40 × 0.30 × 0.25 mm

Data collection

Nonius KappaCCD diffractometer
φ scans
Absorption correction: none
12041 measured reflections
2694 independent reflections
2331 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 30.0°
$h = -10 \rightarrow 12$
$k = 15 \rightarrow 15$
$l = -18 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.115
S = 1.17
2654 reflections
137 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.3395P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S—O4	1.4343 (12)	S—O6	1.4589 (12)
S—O3	1.4554 (12)	S—O5	1.5574 (12)
O4—S—O3	114.11 (8)	O4—S—O5	103.51 (8)
O4—S—O6	113.31 (8)	O3—S—O5	106.72 (7)
O3—S—O6	111.34 (8)	O6—S—O5	107.06 (7)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H11···O2^{i}	0.89	2.03	2.9101 (18)	169
N—H12···O4^{ii}	0.89	1.98	2.8010 (18)	153
N—H13···O6	0.89	2.02	2.8757 (18)	162
O5—H5···O6^{iii}	0.82	1.87	2.6776 (17)	166
O1—H1···O3^{iv}	0.82	1.87	2.6843 (16)	173

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $2-x, 1-y, 1-z$; (iii) $2-x, 1-y, 2-z$; (iv) $x-1, y, z$.

All H atoms were placed geoetrically and treated as riding, with C—H distances of 0.93 Å, O—H distances of 0.82 Å and N—H distances of 0.89 Å, and U_{iso}(H) values of 1.2 times U_{eq} of the carrier atom.

Data collection: *KappaCCD Reference Manual* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR*2002 (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996), *ORTEP*-3 (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank Drs M. Pierrot and M. Giorgi from LBS–UMR 6517, Faculté des Sciences et Techniques de Saint Jérôme, Avenue Escadrille Normandie Niemen, 13397 Marseille Cedex 20, France, for providing diffraction facilities.

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