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Bis(2-carboxyanilinium) sulfate monohydrate

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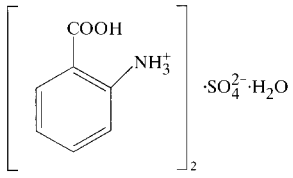
The title compound, $2C_7H_8NO_2^+\cdot SO_4^{2-}\cdot H_2O$, (I), exhibits a complex three-dimensional network of hydrogen bonds, involving all hydrogen donor atoms. A total of ten hydrogen bonds are present in the asymmetric unit, five of which are three-centre hydrogen bonds with one hydrogen donor and two acceptors. The suitability of the compound for possible charge–density study was investigated. As the quality of crystals did not seem sufficient for this purpose, no further experiments were carried out.



(I)

Experimental

Crystals of (I) were grown by very slow evaporation of an ethanol solution of anthranilic acid and sulfuric acid (95% water solution) in an equimolar ratio.

Crystal data

$2C_7H_8O_2^+\cdot SO_4^{2-}\cdot H_2O$
M_r = 390.36
Monoclinic, $P2_1/c$
a = 11.0632 (5) Å
b = 15.8711 (9) Å
c = 9.9597 (4) Å
β = 97.559 (3)°
V = 1733.58 (14) Å^3
Z = 4
D_x = 1.496 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7879 reflections
θ = 1.018–25.028°
μ = 0.239 mm^{-1}
T = 293 (2) K
Plate, translucent colourless
0.4 × 0.3 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer
Method: CCD rotation scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.882, T_{max} = 0.931
14 306 measured reflections
3050 independent reflections
2164 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 25.07°
$h = 0 \rightarrow 13$
$k = -18 \rightarrow 18$
$l = -11 \rightarrow 11$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.140
S = 1.026
3050 reflections
292 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 1.3544P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0053 (14)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N18—H18A···O3^{i}	0.89	2.08	2.901 (4)	153
N18—H18A···O4^{i}	0.89	2.35	3.112 (4)	144
N18—H18B···O12	0.89	1.96	2.696 (4)	140
N18—H18B···O21	0.89	2.33	3.000 (4)	132
N18—H18C···O2	0.89	1.91	2.791 (4)	173
N28—H28A···O4^{ii}	0.89	1.88	2.760 (4)	170
N28—H28B···O21	0.89	1.94	2.687 (4)	141
N28—H28B···O12	0.89	2.28	2.926 (4)	129
N28—H28C···O2^{iii}	0.89	2.01	2.825 (4)	151
N28—H28C···O5^{iii}	0.89	2.56	3.348 (5)	148
O6—H61···O5^{iv}	0.84 (5)	1.86 (5)	2.693 (4)	171 (4)
O6—H62···O4	0.85 (5)	1.94 (5)	2.776 (4)	169 (4)
O11—H11···O6^{ii}	0.93 (6)	1.66 (6)	2.577 (4)	170 (5)
O22—H22···O3	0.90 (2)	1.86 (2)	2.737 (4)	165 (6)
O22—H22···O5	0.90 (2)	2.59 (5)	3.231 (6)	129 (5)

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

During the refinement, all the H atoms were located and refined, with the exception of the carboxyl H atom. The structure was transferred to the *JANA* (Petricek & Dusek, 2000) crystallographic computing system and the section of the difference Fourier map through the carboxylic acid group was drawn, on which the remaining H22 atom was located. Having experimental evidence that the H22 atom was present in the crystal, the atom was set to a calculated positionand and refined in *SHELXL* using two constraints: (i) the O22—H22 distance was kept at 0.9 Å, within an s.u. of 0.02 Å; (ii) the carboxylic acid group C27—O21—O22—H22 was kept planar within an s.u. of 0.1 Å. During the refinement, some H atoms of the $-NH_3^+$ groups were moving slowly towards the parent N18 and N28 atoms. These groups were constrained to idealized geometry with tetrahedral angles, fixed N—H distances, free rotation around the C—N bond and refined isotropic displacement parameters for H atoms.

Data collection: *COLLECT* (Nonius, 1999); cell refinement and data reduction: *HKL SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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