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2-Ammonio-3-phenylpropanoic acid–monohydrogen sulfate–2-ammonio-3-phenylpropanoate (1/1/1)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.011 Å
R factor = 0.075
wR factor = 0.163
Data-to-parameter ratio = 13.0

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

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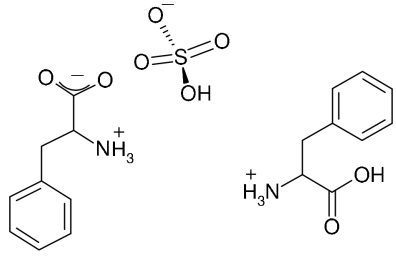
The title compound, $C_9H_{12}NO_2^+\cdot HSO_4^-\cdot C_9H_{11}NO_2$, consists of a neutral 2-ammonio-3-phenylpropanoate zwitterionic molecule, a 2-ammonio-3-phenylpropanoic acid cation, and a hydrogen sulfate anion. All the O and N atoms in the compound contribute to hydrogen bonds, leading to the formation of sheets parallel to the *ab* plane.

Comment

The title compound, (I) (Fig. 1), consists of a neutral 2-ammonio-3-phenylpropanoate zwitterionic molecule, a 2-ammonio-3-phenylpropanoic acid cation, and a hydrogen sulfate anion. The bond lengths and angles of the 2-ammonio-3-phenylpropanoate moiety are in normal ranges, similar to those observed previously (You *et al.*, 2003; Liang *et al.*, 2002). The dihedral angle between the C1–C6 and C10–C15 phenyl rings is 87.5 (7)°, while that formed by the O1/C9/O2 carboxylate group and the C1–C6 phenyl ring is 138.9 (7)°. In the 2-ammonio-3-phenylpropanoic acid cation, the torsion angles C1—C7—C8—C9 and C1—C7—C8—N1 are 90.6 (6) and −150.6 (6)°, respectively. In the neutral 2-ammonio-3-phenylpropanoate molecule, the torsion angles C10—C16—C17—C18 and C10—C16—C17—N2 are 160.5 (6) and −79.0 (6)°, respectively. Atom C8 deviates from the plane of the C1–C6 phenyl ring by 1.243 (7) Å, whereas atom C17 deviates from the plane of the C10–C15 phenyl ring by 1.289 (7) Å.



(I)

The hydrogen sulfate anion is linked to the neutral 2-ammonio-3-phenylpropanoate molecule (Fig. 1) *via* an O6—H6···O3^{i} hydrogen bond (see Table 1 and Fig. 2).

In the crystal structure, the three moieties are connected by O—H···O and N—H···O hydrogen bonds (Table 1 and Fig. 2), and are stacked along the *b* axis.

Experimental

2-Amino-3-phenylpropane-1,1-diol and sulfuric acid were available commercially and were used without further purification. A mixture

of 2-amino-3-phenylpropane-1,1-diol (0.1 mmol, 16.5 mg) and sulfuric acid (0.1 mmol, 10.0 mg) was dissolved in distilled water (12 ml). The mixture was stirred for 30 min at room temperature to give a clear colorless solution. After allowing the resulting solution to stand in air for 8 d, colorless block-like crystals of (I) had formed at the bottom of the vessel on slow evaporation of the water (yield 71.2%). Analysis found: C 50.3, H 5.7, N 6.4%; calculated for $C_{18}H_{24}N_2O_8S$: C 50.5, H 5.6, N 6.5%.

Crystal data

$C_9H_{12}NO_2^+\cdot HSO_4^-\cdot C_9H_{11}NO_2$
M_r = 428.45
Monoclinic, $P2_1$
a = 13.439 (3) Å
b = 4.878 (3) Å
c = 15.483 (3) Å
β = 97.05 (3)°
V = 1007.4 (4) Å^3
Z = 2
D_x = 1.412 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 512 reflections
θ = 2.7–16.4°
μ = 0.21 mm^{-1}
T = 293 (2) K
Block, colorless
0.27 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.946, T_{max} = 0.963
5540 measured reflections
3478 independent reflections
2086 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 26.0°
$h = -15 \rightarrow 16$
$k = -5 \rightarrow 5$
$l = -17 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.163
S = 0.95
3478 reflections
268 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Absolute structure: Flack (1983); 1260 Friedel pairs
Flack parameter = 0.15 (19)

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O3^{i}	0.83 (6)	1.69 (6)	2.519 (5)	176 (8)
N2—H2C···O5^{ii}	0.89	1.98	2.828 (6)	160
N2—H2B···O5^{iii}	0.89	2.17	3.007 (6)	157
N2—H2A···O5	0.89	2.04	2.923 (6)	175
N1—H1C···O2^{iv}	0.89	2.57	3.047 (6)	114
N1—H1C···O1^{v}	0.89	2.15	3.005 (6)	162
N1—H1B···O4^{iii}	0.89	1.91	2.730 (6)	153
N1—H1A···O7^{iii}	0.89	2.02	2.905 (6)	172
O1—H1···O7^{vi}	0.82	1.73	2.543 (5)	171

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

Atom H6 was located in a difference Fourier map and refined, with the U_{iso}(H) value fixed at 0.08 Å^2. The other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H, O—H and N—H distances of 0.93–0.98, 0.82–0.83 and 0.89 Å, respectively, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(O/N).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

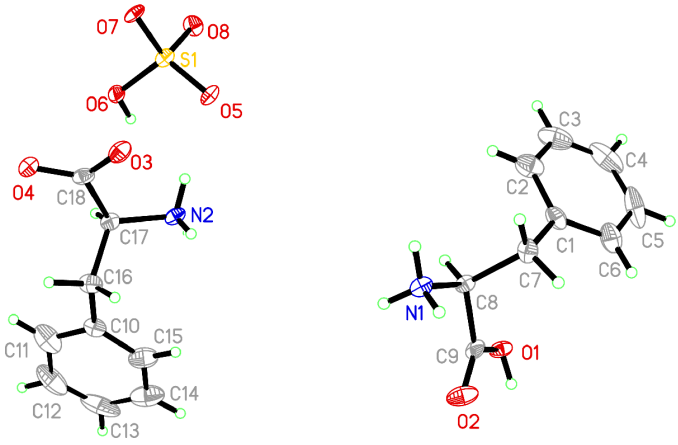


Figure 1
The asymmetric unit of the title compound, (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

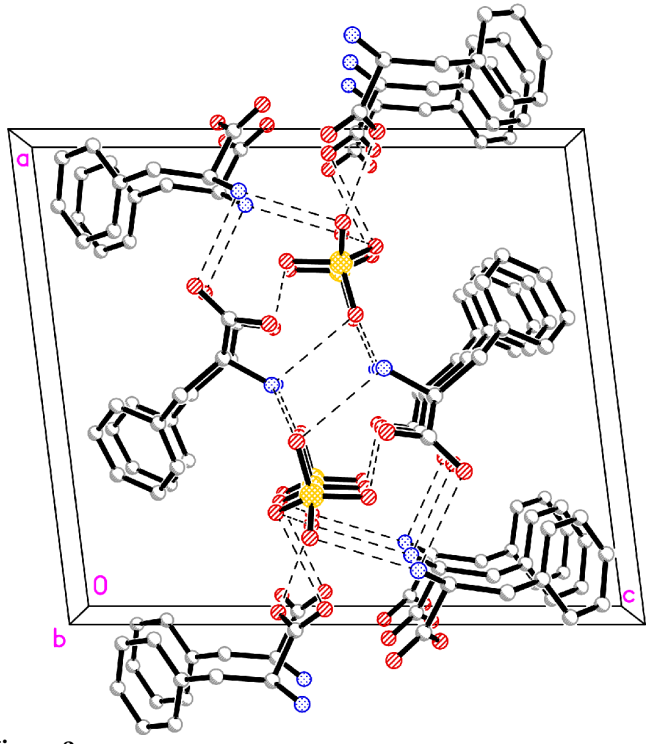


Figure 2
Crystal packing of (I), viewed along the b axis. O···O contacts are shown as dashed lines. H atoms have been omitted for clarity.

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References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Liang, H., Yu, Q. & Hu, R.-X. (2002). *Transition Met. Chem.* **27**, 454–457.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
You, Z.-L., Zhu, H.-L., Liu, W.-S. & Tan, M.-Y. (2003). *Acta Cryst.* E**59**, o1920–o1922.