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Bis(D-phenylglycinium) sulfate monohydrate

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Key indicators

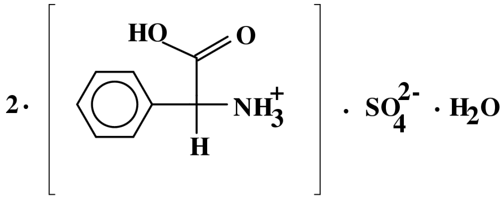
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.043
wR factor = 0.133
Data-to-parameter ratio = 7.1

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

In the title compound, $2C_8H_{10}NO_2^+\cdot SO_4^{2-}\cdot H_2O$, the cations exist as two crystallographically independent molecules in the asymmetric unit. In the crystal, these molecules are linked by the sulfate ion through strong O—H···O hydrogen bonds; the phenylglycinium cations, sulfate anion and water molecule are held together by N—H···O and O—H···O hydrogen bonds.

Comment

D-Phenylglycine is an important starting material in the production of semisynthetic penicillins and cephalosporins and its derivatives are used in the synthesis of antitumor drugs and other pharmacological applications (Satyam *et al.*, 1996; Jayasinghe *et al.*, 1994). The crystal structure of D-phenylglycine hydrochloride (Ravichandran *et al.*, 1998) has been reported. In the present study, the phenylglycine reacted with sulfuric acid was studied.



(I)

The asymmetric unit of the title compound, (I), consists of two crystallographically independent protonated phenylglycinium cations, a sulfate anion and a water molecule. In the phenylglycinium molecules, the O1—C11—C12—N11 and O3—C21—C22—N21 torsion angles of 25.0 (5) and 30.7 (5)°, respectively, show that the orientation of the carboxyl group is influenced by the phenyl substitution at the C^α atoms. In α-glycine (Marsh, 1958) and diglycine hydrochloride (Natarajan *et al.*, 1992), the O1—C11—C12—N11 values are 19.1 and 0.3°, and 16.5°, respectively. Molecular aggregation gives a hydrophilic zone along [101] and a hydrophobic zone along [202].

All the O atoms of the sulfate ion are involved in hydrogen bonding. One of the O atoms (O1*D*), as acceptor, links two crystallographically independent phenylglycinium molecules through strong hydrogen bonds [2.610 (5) and 2.578 (4) Å], resulting in an increased S1—O1*D* bond distance [1.505 (3) Å]. A bifurcated hydrogen bond is observed between an amino N atom and sulfate O atoms (Jeffrey & Saenger, 1991). A zigzag (*Z*1) head-to-tail sequence is observed, since N11—H11*B*···O1$(-x+2,\ y+\frac{1}{2},\ -z)$ and N21—H21*A*···O3$(-x+1,\ y-\frac{1}{2},\ -z+1)$ connect two 2_1-related amino acids (Vijayan, 1988).

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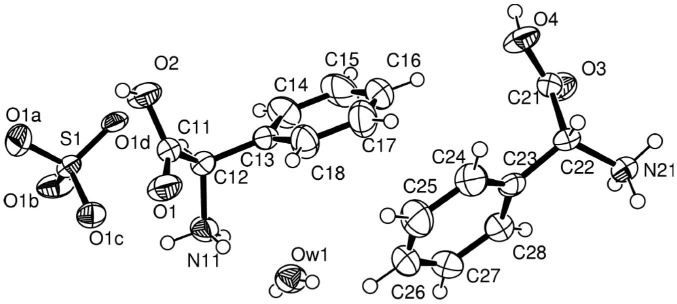

Figure 1
The structure of (I) with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

Experimental

The title compound was crystallized by slow evaporation of an aqueous solution of D-phenylglycine and sulfuric acid in a stoichiometric ratio of 2:1. Colourless needle-shaped transparent crystals were obtained.

Crystal data

$2C_8H_{10}NO_2^+ \cdot SO_4^{2-} \cdot H_2O$
$M_r = 418.42$
Monoclinic, $P2_1$
$a = 12.3201\ (12)$ Å
$b = 5.9377\ (15)$ Å
$c = 14.2908\ (16)$ Å
$\beta = 111.369\ (10)^\circ$
$V = 973.5\ (3)$ Å^3
$Z = 2$
$D_x = 1.427$ Mg m^{-3}

$D_m = 1.429$ Mg m^{-3}
D_m measured by flotation
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 8.3$–13.6°
$\mu = 0.22$ mm^{-1}
$T = 293\ (2)$ K
Needle, colourless
$0.25 \times 0.20 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.949$, $T_{\max} = 0.968$
1968 measured reflections
1877 independent reflections
1778 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.044$
$\theta_{\max} = 25.0^\circ$
$h = -14 \rightarrow 13$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.133$
$S = 1.07$
1877 reflections
263 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1005P)^2 + 0.3547P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}
Absolute structure: Flack (1983), 91 Friedel pairs
Flack parameter = −0.05 (14)

Table 1
Selected geometric parameters (Å, °).

S1—O1*A*	1.451 (4)	O1—C11	1.218 (5)
S1—O1*B*	1.460 (4)	O2—C11	1.305 (5)
S1—O1*C*	1.470 (3)	O3—C21	1.196 (6)
S1—O1*D*	1.505 (3)	O4—C21	1.312 (5)
O1—C11—C12—N11	25.0 (5)	O3—C21—C22—N21	30.7 (5)
N11—C12—C13—C18	−77.3 (5)	N21—C22—C23—C28	−41.6 (5)
C11—C12—C13—C18	43.6 (5)	C21—C22—C23—C28	79.6 (4)

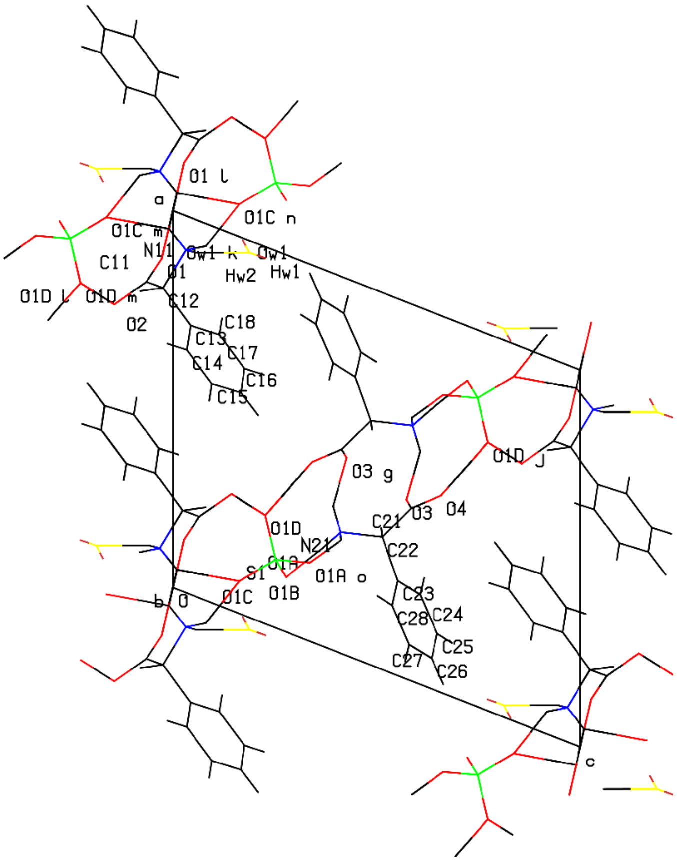

Figure 2
Packing diagram of the molecules viewed down the *b* axis.

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
O2—H2$\cdots$O1*D*$^{\rm i}$	0.82	1.79	2.610 (5)	174
O4—H4$\cdots$O1*D*$^{\rm ii}$	0.82	1.76	2.578 (4)	175
N11—H11*A*$\cdots$O*W*1$^{\rm iii}$	0.89	1.95	2.680 (6)	139
N11—H11*B*$\cdots$O1$^{\rm iv}$	0.89	2.25	2.877 (6)	128
N11—H11*B*$\cdots$O1*C*$^{\rm v}$	0.89	2.30	2.969 (5)	132
N11—H11*C*$\cdots$O1*C*$^{\rm vi}$	0.89	2.25	3.049 (5)	149
N21—H21*A*$\cdots$O3$^{\rm ii}$	0.89	2.30	2.951 (5)	130
N21—H21*B*$\cdots$O1*A*	0.89	1.97	2.761 (5)	147
N21—H21*C*$\cdots$O1*B*$^{\rm vii}$	0.89	1.78	2.674 (5)	178
O*W*1—H*W*2$\cdots$O1*C*$^{\rm viii}$	0.84 (9)	1.95 (9)	2.771 (5)	164 (7)
O*W*1—H*W*1$\cdots$O1*B*$^{\rm ix}$	0.94 (7)	1.88 (8)	2.798 (6)	167 (7)

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

The H atoms of the water molecule were located from difference Fourier maps and refined, while all the other H atoms were fixed by geometric constraints using *HFIX*. 91 Friedel pairs were used to determine the Flack parameter.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.

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