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Diglycinium sulfate

Aouatef Cherouana, Nourredine Benali-Cherif,* Lamia Bendjeddou and Hocine Merazig

Laboratoire de Chimie Moléculaire, du Contrôle de l'Environnement et des Mesures Physico-Chimiques, Faculté des Sciences, Département de Chimie, Université Mentouri de Constantine, 25000 Constantine, Algeria

Correspondence e-mail: benalicherif@hotmail.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.097
Data-to-parameter ratio = 12.2

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

The crystal structure of diglycinium sulfate (DGS), $2C_2H_6NO_2^+ \cdot SO_4^{2-}$, consists of two layers of glycinium and sulfate groups inserted between a layer of glycinium cations linked by strong hydrogen bonds. The two glycinium cations have different conformations, *viz*. *E* for glycine A and *Z* for glycine B.

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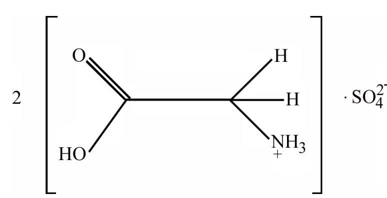
Comment

Many glycine salts and adducts exhibit interesting dielectric properties, the best known compound of this family being TGS (triglycine sulfate), which orders ferroelectrically below 322 K (Matthias *et al*., 1956). In addition to their importance in the field of new materials chemistry (Siegel *et al*., 1998; Baker *et al*., 1992), hybrid compounds are of interest because of their electrical, magnetic and optical properties (Kagan *et al*., 1999; Hill, 1998). The title compound, diglycinium sulfate (DGS), results from a search for new organic–inorganic hybrid materials (Benali-Cherif, Cherouana *et al*., 2002; Benali-Cherif, Abouimrane *et al*., 2002; Benali-Cherif, Bendheif *et al*., 2002; Benali-Cherif, Benguedouar *et al*., 2002.). The asymmetric unit of DGS contains two monoprotonated glycine molecules ($C_2H_7NO_2^+$) and one sulfate ion (SO_4^{2-}).



DGS

The mean bond lengths and angles in the SO_4^{2-} group are 1.472 Å and 109.5°, respectively, showing a normal tetrahedral geometry for the S atom. Interatomic distances in the glycinium cations compare well with distances observed in diglycine sulfate monohydrate (Cano & Martinez-Carrera, 1974). Although their carboxy skeletons are both planar, atom N1*B* is displaced from the plane by 0.170 (15) Å, whereas atom N1*A* is displaced by 0.075 (15) Å. The relevant torsion angles of the glycinium cations [O1*A*—C2*A*—C1*A*—N1*A* = 176.71 (15)° and N1*B*—C2*B*—C1*B*—O1*B* = 23.9 (7)°] indicate different conformations for the two glycinium cations in the asymmetric unit, *viz*. *E* for glycine A and *Z* for glycine B; this difference in conformation is not observed in diglycine selenate (Olejnik & Lukaszewicz, 1975). The crystal structure is composed of two layers of glycine B ions and sulfate groups inserted between layers of glycine A along their stacking direction (*b* axis). The layers are linked together by an intricate network of hydrogen-bond interactions. The strongest of

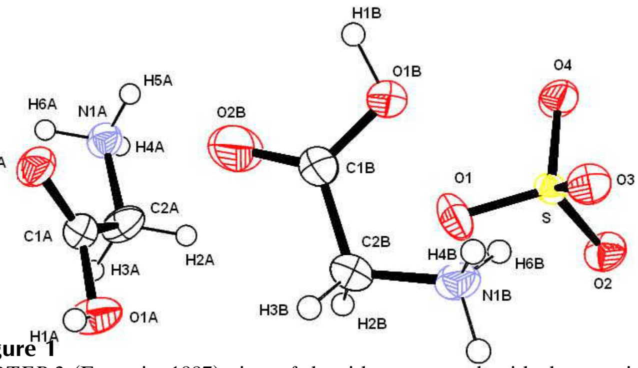


Figure 1
ORTEP-3 (Farrugia, 1997) view of the title compound, with the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Only one disorder component is shown.

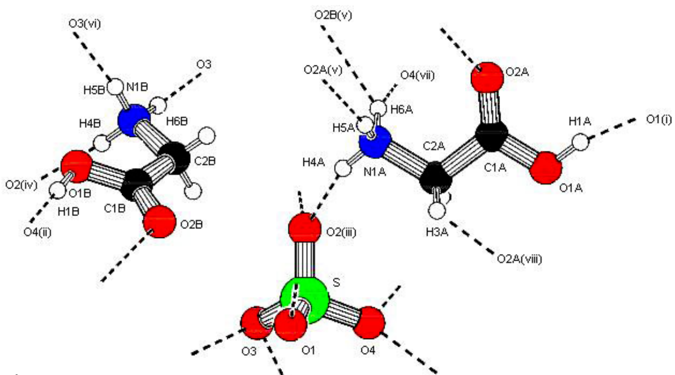


Figure 2
PLUTON (Spek, 1990) view of the title compound, showing the immediate hydrogen-bonding environment of the anion and cations.

these bonds involve atoms O2*A* and O2*B* of the glycinium cations (*A* and *B*) as donors and O atoms of sulfate anions as acceptors; details of hydrogen bonds are given in Table 1. Atom N1*B* forms three hydrogen bonds with sulfate anions. Weak hydrogen bonds are observed in the glycine A layers, between N and O atoms, and between C and O atoms.

Experimental

Colorless single crystals of DGS were obtained by slow evaporation, at room temperature, of an equimolar solution of glycine and sulfuric acid.

Crystal data

$2C_2H_6NO_2^+ \cdot SO_4^{2-}$
$M_r = 248.23$
Orthorhombic, *Pbca*
$a = 8.9350$ (4) Å
$b = 10.2770$ (3) Å
$c = 21.7640$ (3) Å
$V = 1998.48$ (11) Å^3
$Z = 8$
$D_x = 1.65$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12129 reflections
$\theta = 3.6$–$26.4°$
$\mu = 0.35$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
$0.5 \times 0.4 \times 0.3$ mm

Data collection

Nonius KappaCCD diffractometer
φ scans
Absorption correction: none
12129 measured reflections
1997 independent reflections
1844 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$
$\theta_{max} = 26.4°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.098$
$S = 1.10$
1997 reflections
164 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0331P)^2 + 0.3591P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1*A*—H1*A*$\cdots$O1^{i}	0.82	1.77	2.567 (2)	164
O1*B*—H1*B*$\cdots$O4^{ii}	0.88 (3)	1.70 (3)	2.573 (2)	172 (3)
N1*A*—H4*A*$\cdots$O2^{iii}	0.89	1.92	2.762 (2)	156
N1*B*—H4*B*$\cdots$O2^{iv}	0.89	1.96	2.838 (2)	170
N1*A*—H5*A*$\cdots$O2*A*^{v}	0.89	2.13	2.958 (2)	154
N1*B*—H5*B*$\cdots$O3^{vi}	0.89	1.96	2.851 (2)	175
N1*A*—H6*A*$\cdots$O21*B*^{v}	0.89	2.48	3.018 (5)	119
N1*A*—H6*A*$\cdots$O22*B*^{v}	0.89	2.56	3.177 (6)	127
N1*A*—H6*A*$\cdots$O4^{vii}	0.89	2.03	2.805 (2)	145
N1*B*—H6*B*$\cdots$O3	0.89	1.93	2.813 (2)	170
C2*A*—H3*A*$\cdots$O2*A*^{viii}	0.97	2.51	3.264 (2)	134

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

In the initial refinement of the title compound, atom O2*B* showed high anisotropy of apparent thermal motion normal to the carboxyl plane. The final refinement was carried out with a model in which atoms O2*B* and C1*B* each have two alternative sites with equal occupancy, to simulate a disorder that occurs by a twist of the C1*B*—O2*B* arm. All H atoms (except H1*B*, which was refined isotropically) were then fixed at geometrically determined positions. Riding isotropic displacement parameters were used for all H atoms.

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*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLUTON* (Spek, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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