for $(n,\pi^*)$, <3.0 Å, 0, 90, 180°. The measured values for the closest S···$\beta$H contact in compound (1) are: 2.71 Å, 13, 70, 97°. The S···H contact is shorter than the sum of van der Waals radii (3.00 Å), very favourable for hydrogen abstraction, with the $\omega$ angular parameter more favourable for abstraction *via* $(n,\pi^*)$. Compounds studied previously which undergo photochemical reaction *via* $(\pi,\pi^*)$ have average $\gamma$H-abstraction parameters of about 3.04 Å, 51, 53, 122° (Fu *et al.*, 1997).

## Experimental

The title compound was synthesized according to the procedure of Couture *et al.* (1981).

*Crystal data*

$C_{18}H_{17}NS$
$M_r$ = 279.40
Orthorhombic
*Pbca*
$a$ = 23.106 (13) Å
$b$ = 16.445 (12) Å
$c$ = 7.985 (7) Å
$V$ = 3034 (7) Å$^3$
$Z$ = 8
$D_x$ = 1.223 Mg m$^{-3}$
$D_m$ not measured
Mo $K\alpha$ radiation
$\lambda$ = 0.7107 Å
Cell parameters from 24 reflections
$\theta$ = 5.8–13.0°
$\mu$ = 0.193 mm$^{-1}$
$T$ = 294 K
Prism
0.50 × 0.25 × 0.20 mm
Purple

*Data collection*

Rigaku AFC-6*S* diffractometer
$\omega$–2$\theta$ scans
Absorption correction: $\psi$ scans (North *et al.*, 1968)
$T_{min}$ = 0.787, $T_{max}$ = 0.962
4429 measured reflections
4429 independent reflections
1485 reflections with $I > 3\sigma(I)$
$\theta_{max}$ = 30.05°
$h = 0 \rightarrow 32$
$k = 0 \rightarrow 23$
$l = -11 \rightarrow 0$
3 standard reflections
every 200 reflections
intensity decay: 3.1%

*Refinement*

Refinement on $F^2$
$R(F)$ = 0.063
$wR(F^2)$ = 0.206
$S$ = 1.225
4429 reflections
181 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + 0.00123(F_o^2)^2]$
$(\Delta/\sigma)_{max}$ = 0.0002
$\Delta\rho_{max}$ = 0.38 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.37 e Å$^{-3}$
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—C1 | 1.619 (3) | C15—C18 | 1.437 (5) |
| N1—C18 | 1.140 (4) | | |
| S1—C1—C2 | 124.4 (2) | C2—C1—C6 | 118.1 (3) |
| S1—C1—C6 | 117.5 (2) | N1—C18—C15 | 178.3 (4) |

H atoms were placed in calculated sites, with C—H = 0.95 Å and $U$(H) = 1.2$U$(bonded C).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1388). Services for accessing these data are described at the back of the journal.

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# Mephentermine Hemisulfate Monohydrate: an Adrenergic Agent

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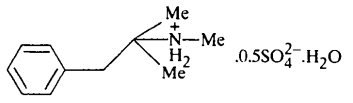
## Abstract

The title molecule, a hydrated hemisulfate salt of ethyl(2-methyl-1-phenyl-2-propyl)ammonium, $C_{11}H_{18}N^+.0.5SO_4^{2-}.H_2O$, consists of a phenethylamine skeleton in which the N atom is protonated. There are two molecules in the asymmetric unit, with the S atom of the $SO_4^{2-}$ ion lying on a pseudo-twofold axis. The ethylamine side chain is in an extended conformation in both the symmetry-independent molecules. The distance of the N atom from the centre of the benzene ring is 5.1 Å for molecule 1 and 5.3 Å for molecule 2. The packing is stabilized by N—H···O and O—H···O hydrogen bonds.

## Comment

Compounds having a phenethylamine backbone are found to be potent adrenergic drugs. Mephentermine belongs to this class of drug molecules and is clinically used as a vasopressor. The three-dimensional structure of this compound has been determined as its hydrated hemisulfate salt, (I), and is compared with those of other similar drug molecules.




(I)

The interatomic bond lengths and angles in (I) are similar in the two molecules in the asymmetric unit and show no significant deviations from the average model values (Herbert, 1979) obtained by averaging bond lengths and angles for 34 similar adrenergic compounds.

The conformation of the ethylamine side chain with respect to the benzene ring is usually described by the torsion angles C2—C1—C7—C8 ($\tau 1$) and C1—C7—C8—N ($\tau 2$). The corresponding angles of $\tau 1$ = −94.9 (9) and $\tau 2$ = 175.2 (7)° for molecule 1, and $\tau 1$ = −91.7 (9) and $\tau 2$ = −175.3 (8)° for molecule 2, indicate that both molecules have a perpendicular *trans* conformation. A study of these torsion angles in other adrenergic agents like *p*-hydroxyephedrine hydrochloride (Dattagupta *et al.*, 1981), synephrine monohydrogen phosphate monohydrate (Dattagupta *et al.*, 1982), L-phenylephrine hydrochloride (Bhaduri *et al.*, 1983), 2,2′-di-*N*-methylamino-1,1′-di-*p*-hydroxyphenyldiethylene ether dihydrobromide (Mukhopadhyay & Dattagupta, 1988) also show similar values. This indicates that a maximally extended ethylamine side chain approximately perpendicular to the benzene ring may be the receptor-preferred conformation for this class of drugs. The distance of the amino N atom from the centre of the benzene ring is 5.1 Å in molecule 1 and 5.3 Å in molecule 2. This distance seems to be fairly constant in similar biologically active amines and appears to be of significance for sympathomimetic activity.

The protonated N atoms, all the O atoms of the $SO_4^{2-}$ ion and the water O atoms participate in hydrogen bonding.

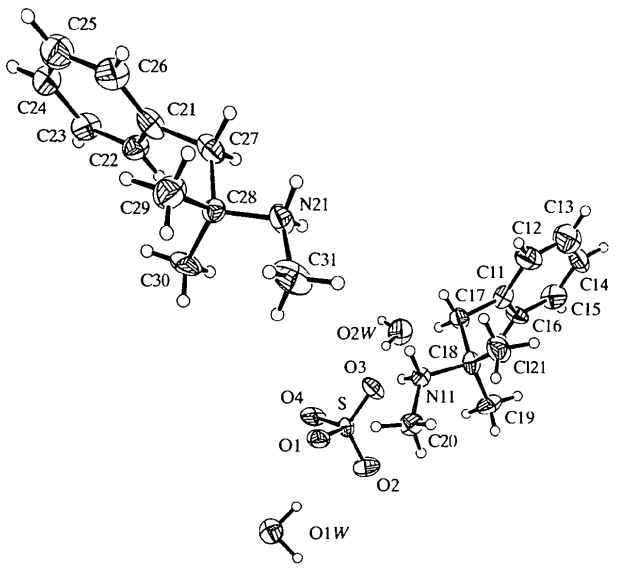



Fig. 1. *ORTEX* (McArdle, 1993) drawing of molecules 1 and 2 of the title salt with the atom-numbering scheme. Displacement ellipsoids are at the 40% probability level.

## Experimental

The title compound was obtained commercially from the Sigma Chemical Company.

*Crystal data*

$C_{11}H_{18}N^+.0.5SO_4^{2-}.H_2O$
$M_r$ = 230.31
Monoclinic
$P2_1/c$
$a$ = 30.150 (10) Å
$b$ = 6.644 (2) Å
$c$ = 12.685 (7) Å
$\beta$ = 102.60 (4)°
$V$ = 2479.8 (18) Å$^3$
$Z$ = 8
$D_x$ = 1.234 Mg m$^{-3}$
$D_m$ = 1.213 Mg m$^{-3}$
$D_m$ measured by flotation in a benzene–bromoform mixture
Cu $K\alpha$ radiation
$\lambda$ = 1.54180 Å
Cell parameters from 25 reflections
$\theta$ = 9–45°
$\mu$ = 1.475 mm$^{-1}$
$T$ = 293 (2) K
Needle
0.6 × 0.3 × 0.3 mm
Colourless

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (North *et al.*, 1968)
$T_{min}$ = 0.602, $T_{max}$ = 0.642
2581 measured reflections
2524 independent reflections
2377 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027
$\theta_{max}$ = 72.73°
$h$ = 0 → 37
$k$ = 0 → 8
$l$ = −15 → 13
3 standard reflections every 200 reflections
intensity decay: not significant

*Refinement*

Refinement on $F^2$
$R(F)$ = 0.051
$wR(F^2)$ = 0.160
$S$ = 1.224
2524 reflections
276 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1045P)^2 + 0.7904P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.090
$\Delta\rho_{max}$ = 0.495 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.301 e Å$^{-3}$
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0036 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

The title salt crystallized in the monoclinic system. Precession and Weissenberg photographs indicated a high degree of pseudo-C-centering. The structure was finally solved in space group $P2_1/c$. Attempts to solve and refine the structure in $C2/c$ led to an $R$ factor of 26%. The S atom of the sulfate ion sits on the pseudo-twofold axes generated by the non-crystallographic C-centering. The phenyl C atoms for both the molecules in the asymmetric unit were regularized with C—C bond lengths of 1.39 Å. The phenyl rings were refined as rigid groups until the last few cycles of refinement. Due to very high correlation between the two molecules of the asymmetric unit, it was difficult to achieve convergence in the refinement and so damping was used during refinement. All the H atoms, except those of the water molecule, were placed in geometrically calculated positions, with an average C—H distance of 0.953 Å and an average N—H distance of 0.90 Å. They were included in the refinement but were restrained to ride on their parent atoms. The isotropic displacement parameters of the H atoms were held fixed to $1.2U_{eq}$ of their riding atoms. The H atoms of the water molecules were located from difference Fourier maps and kept completely fixed. All hydrogen-bond calculations were carried out using *PARST* (Nardelli, 1983).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEX2.1a* (McArdle, 1993). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1069). Services for accessing these data are described at the back of the journal.

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# D-Phenylglycine Hydrochloride

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## Abstract

In the title compound, $C_8H_{10}NO_2^+.Cl^-$, the hydrochloride of a ring-substituted amino acid, the amino N atom is protonated. As in other amino acids, this atom lies nearly in the plane of the carboxyl group, indicating thereby that the orientation of the group is unaffected by the phenyl substitution at the $\alpha$-C atom. The crystal structure, consisting of alternating layers of hydrophobic and hydrophillic zones, is stabilized by hydrogen bonds involving mostly the Cl atom.

## Comment

D-Phenylglycine is an important starting material in the production of $\beta$-lactams such as semisynthetic penicillins and cephalosporins. Many phenylglycine derivatives are also well known for their use in the synthesis of antitumor drugs and other pharmacological applications (Satyam *et al.*, 1996, Jayasinghe *et al.*, 1994).

The amino N atom in the title molecule, (I), is protonated. The bond lengths and angles in the structure compare well with those found in $\alpha$-glycine (Marsh, 1958), diglycine hydrochloride (Hahn & Buerger, 1957; Faamau & Tiekink, 1993), the crown ether inclusion complex of (*R*)-phenylglycine methyl ester (Goldberg, 1977) and (*R*)-(−)-1-phenylglycinium hydrogen squarate monohydrate (Angelova *et al.*, 1996). The torsion angle N—C7—C8—O1, which indicates the relative orientation of the carboxyl group and the amino N atom, is 18.9 (5)°. This value is close to the corresponding values of 19.1 and 16.3° in $\alpha$-glycine (Marsh, 1958) and diglycine hydrochloride (Hahn & Buerger, 1957; Faamau & Tiekink, 1993), respectively, thereby indicating that the orientation of the carboxyl group is not influenced by the phenyl substitution at the $\alpha$-C atom. The orientation of the phenyl ring, as described by the torsion angle C3—C4—C7—N of 129.2 (3)°, is close to the corresponding value (137.6°) in (*R*)-phenylglycine methyl ester (Goldberg, 1977) and that (119.9°) in (*R*)-(−)-1-phenylglycinium hydrogen squarate monohydrate (Angelova *et al.*, 1996).