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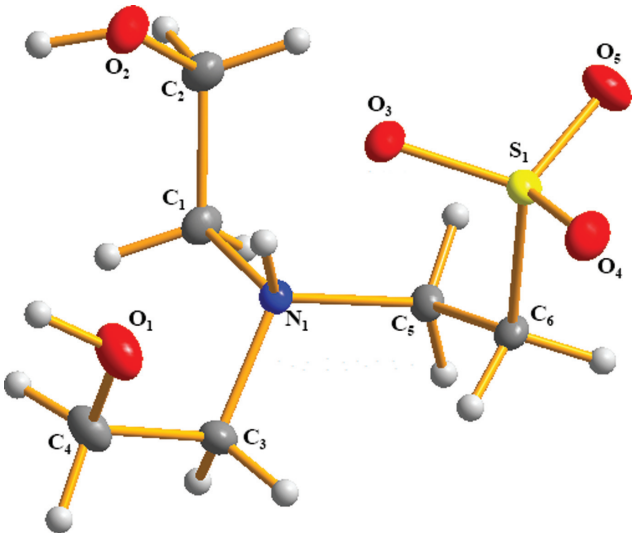
Crystal structure of 2-(bis(2-hydroxyethyl) ammonio)ethane-1-sulfonate, $C_6H_{15}NO_5S$



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Abstract

$C_6H_{15}NO_5S$, monoclinic, *Cc* (no. 9), $a = 15.167(5)$ Å, $b = 6.422(5)$ Å, $c = 10.969(5)$ Å, $\beta = 117.407(5)°$, $V = 948.5(9)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0278$, $wR_{ref}(F^2) = 0.0755$, $T = 293(2)$ K.

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A part of the molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

An ethanolic solution of 2-oxoethanesulfonic acid (10 mmol) was added to a solution of bis(2-hydroxyethyl)ammonium (10 mmol) in ethanol and the solution was refluxed for 6 hours. The reaction mixture was then cooled to room temperature and $MgSO_4$ was added to remove some water. The $MgSO_4$ was filtered off and the filtrate was dried overnight under reduced pressure. Crystals were grown through a slow evaporation of an ethanol solution at 298 K.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.23 × 0.20 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.33 mm^{-1}
Diffractometer, scan mode:	CCD, φ and ω
θ_{max}, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3658, 1757, 0.016
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1667
$N(param)_{refined}$:	124
Programs:	Bruker [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.9420(2)	0.5922(4)	0.7514(3)	0.0362(6)
H1A	0.8701	0.5826	0.7037	0.043*
H1B	0.9663	0.4877	0.8233	0.043*
C2	0.9824(3)	0.5475(5)	0.6518(3)	0.0421(7)
H2A	1.0538	0.5675	0.6976	0.051*
H2B	0.9692	0.4033	0.6228	0.051*
C3	0.8951(2)	0.8925(5)	0.8525(3)	0.0365(6)
H3A	0.9252	1.0009	0.9209	0.044*
H3B	0.8718	0.7837	0.8920	0.044*
C4	0.8084(2)	0.9816(6)	0.7277(4)	0.0481(8)
H4A	0.7719	0.8708	0.6643	0.058*
H4B	0.7636	1.0530	0.7545	0.058*
C5	1.0731(2)	0.7966(4)	0.9378(3)	0.0313(6)
H5A	1.1160	0.7076	0.9168	0.038*
H5B	1.0675	0.7356	1.0149	0.038*
C6	1.1202(2)	1.0105(4)	0.9786(3)	0.0334(6)
H6A	1.1752	1.0040	1.0706	0.040*
H6B	1.0717	1.1079	0.9797	0.040*
N1	0.97143(16)	0.8041(3)	0.8150(2)	0.0270(5)
H1	0.9754	0.8967	0.7467	0.032*
O1	0.84571(19)	1.1214(4)	0.6648(3)	0.0604(7)
O2	0.93950(19)	0.6780(4)	0.5350(2)	0.0493(6)
O3	1.08107(16)	1.0798(4)	0.7281(2)	0.0436(5)
O4	1.19202(19)	1.3188(4)	0.9014(2)	0.0511(6)
O5	1.24777(17)	0.9726(4)	0.8840(3)	0.0603(7)
S1	1.16424(4)	1.10258(10)	0.86372(4)	0.03323(17)
H2	0.800(2)	1.128(5)	0.5742(14)	0.050*
H3	0.8759(13)	0.642(6)	0.485(4)	0.050*

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Experimental details

The U_{iso} values of the C-bound hydrogen atoms were set to $1.2U_{eq}$(C). Hydrogen atoms of water molecules were located from electron density map. The absolute structure was established by refinement of the Flack parameter (−0.009(11) from 1458 selected quotients) using Parsons' method [4]. A Flack parameter of 0.012(15) was obtained.

Comment

The research of coordination polymers (CPs) are significant and unparalleled for their diversified, designable and tailorable structures as well as unique chemical and physical properties [5–7]. Thus the design and synthesise of organic ligands is of great significance. Sulfur-rich compounds have been explored in various ways as ligands in coordination chemistry [8]. When the sulfo-groups are introduced to the system, they can coordinate with some metal ions directly in intriguing ways [9, 10]. Thus, it would be valuable to explore novel sulfo-based ligands.

The asymmetric unit contains one molecular unit of the title compound (see the figure). The bond distances and bond angles are in normal ranges. The N1—C5—C6—S1 torsion angle is −74.9(3)°. The bond lengths of C—C, C—N and C—O are in the range of 1.505(4)–1.518(4) Å, 1.500(3)–1.511(3) Å and 1.401(4)–1.414(4) Å, respectively. The bond length of C—S is 1.776(3) Å. In addition, there exist two O—H···O intermolecular hydrogen bonds between sulfo and hydroxyl groups: O1—H2···O4$^{¢i}$ [H2···O4 = 1.880(17) Å, O1···O4 = 2.780(3) Å with angle at H2 = 168° for symmetry code: (¢i) = −0.5 + x, 0.5 − y, −0.5 + z] and O2—H3···O5$^{¢¢}$ [H3···O5 = 1.89(2) Å, O2···O5 = 2.775(3) Å with angle at H3 = 172° for symmetry code: (¢¢) = −0.5 + x, −1.5 + y, −0.5 + z]. In the crystal, the O—H···O hydrogen bonds link together neighboring molecules to form two-dimensional network. Furthermore, there are two intramolecular NH···O hydrogen bonds (N1···O1: 2.759(3) Å and N1···O3: 2.876(3) Å; see the figure).

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