

## Diethylammonium 2,5-dihydroxybenzenesulfonate

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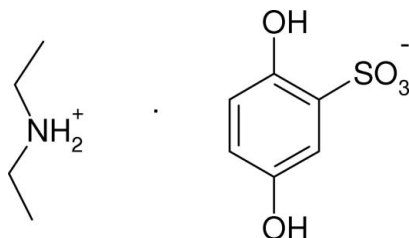
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Key indicators: single-crystal X-ray study;  $T = 291$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.029;  $wR$  factor = 0.079; data-to-parameter ratio = 20.8.

The title compound,  $\text{C}_4\text{H}_{12}\text{N}^+\cdot\text{C}_6\text{H}_5\text{O}_5\text{S}^-$ , also known as ethamsylate, is a molecular salt. Anion-to-anion  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds lead to helices containing  $C(6)$  and  $C(7)$  chains and further bonds of the same type cross-link the chains. Cation-to-anion  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds complete the structure.

### Related literature

For a related structure, see: Solans *et al.* (1982). For background, see: Harrison *et al.* (2007); Hernandez *et al.* (2004); Garay *et al.* (2006). For reference structural data, see: Allen *et al.* (1987). For hydrogen-bonding motifs, see: Bernstein *et al.* (1995).



### Experimental

#### Crystal data

$\text{C}_4\text{H}_{12}\text{N}^+\cdot\text{C}_6\text{H}_5\text{O}_5\text{S}^-$   
 $M_r = 263.31$   
Trigonal,  $P3_2$   
 $a = 8.7006$  (4) Å  
 $c = 14.0732$  (6) Å  
 $V = 922.62$  (7) Å<sup>3</sup>

$Z = 3$   
Mo  $K\alpha$  radiation  
 $\mu = 0.27$  mm<sup>-1</sup>  
 $T = 291$  (2) K  
 $0.40 \times 0.35 \times 0.30$  mm

#### Data collection

Bruker SMART1000 CCD diffractometer  
Absorption correction: multi-scan (SADABS; Bruker, 1999)  
 $T_{\min} = 0.900$ ,  $T_{\max} = 0.926$

7627 measured reflections  
3516 independent reflections  
3307 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.079$   
 $S = 1.08$   
3516 reflections  
169 parameters  
1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.22$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.17$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983), with 1734 Friedel pairs  
Flack parameter: 0.03 (5)

**Table 1**  
Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O3}^{\text{i}}$	0.85 (2)	2.02 (3)	2.8441 (17)	163 (2)
$\text{O2}-\text{H2}\cdots\text{O4}^{\text{ii}}$	0.75 (3)	1.90 (3)	2.6480 (19)	174 (3)
$\text{N1}-\text{H3}\cdots\text{O3}$	0.82 (3)	2.43 (3)	3.084 (2)	138 (2)
$\text{N1}-\text{H3}\cdots\text{O2}^{\text{iii}}$	0.82 (3)	2.49 (3)	3.0691 (19)	129 (2)
$\text{N1}-\text{H4}\cdots\text{O5}^{\text{iv}}$	0.96 (3)	1.94 (3)	2.888 (2)	171 (2)

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

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2077).

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**supplementary materials**

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## Diethylammonium 2,5-dihydroxybenzenesulfonate

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### Comment

The title compound, (I), ethamsylate, or diethylammonium 2,5-dihydroxybenzenesulfonate,  $C_4H_{12}N^+ \cdot C_6H_5O_5S^-$ , is a drug known to possess antihemorrhagic properties. It is said to correct abnormal platelet adhesion, thereby reducing capillary bleeding (Hernandez *et al.*, 2004; Garay *et al.*, 2006).

In continuation of our work on crystal structures of pharmaceutical compounds (Harrison *et al.*, 2007) and in view of the importance of (I), its crystal structure is now reported. The crystal structure of the related bis(diethylammonium) 2,5-dihydroxy-1,4-benzenedisulfonate,  $(C_4H_{12}N^+)_2 \cdot C_6H_4O_2S_2^{2-}$ , was reported earlier (Solans *et al.*, 1982).

Compound (I) formally arises *via* proton transfer from the sulfonic acid group to the secondary amine N atom (Fig. 1) to yield a molecular salt. There are no previous crystal structures containing the 2,5-dihydroxybenzenesulfonate anion, but its individual bond lengths and angles are similar to the corresponding values in the related 2,5-dihydroxy-1,4-benzenedisulfonate (Solans *et al.*, 1982). Overall, the geometrical parameters of the two components in (I) may be regarded as normal (Allen *et al.*, 1987).

Compound (I) crystallizes in the chiral space group  $P3_2$ , with a well defined absolute structure (Flack, 1983). The constituents are achiral, so the enantiomorphic nature of the structure must arise from the packing, and an equal number of enantiomers (crystallizing in  $P3_1$  and  $P3_2$ ) must exist in the bulk sample. The packing is influenced by O—H $\cdots$ O and N—H $\cdots$ O hydrogen bonds (Table 1). Considered in isolation, both the O1—H1 $\cdots$ O3<sup>i</sup> and O2—H2 $\cdots$ O4<sup>ii</sup> bonds (see Table 1 for symmetry codes) lead to helical chains propagating in [001]. The first of these results in a C(6) chain, the second in a C(7) chain (Fig. 2). Together, the O—H $\cdots$ O bonds lead to a three-dimensional array. A strong, near linear N—H $\cdots$ O and a weak, bifurcated N—H $\cdots$ (O,O) hydrogen bond, which link the cation to adjacent anions, complete the structure.

### Experimental

The title compound was obtained as a gift sample from Arvee Chem Pharma, Mysore, India. Crystals of (I) were grown by slow evaporation of an ethanol solution (m.p.: 400 K).

### Refinement

The N- and O-bound H atoms were located in a difference map and their positions were freely refined with  $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$ . The C-bound H atoms were geometrically placed (C—H = 0.93–0.98 Å) and refined as riding with  $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$  or  $1.5U_{eq}(\text{methyl C})$ . The methyl groups were allowed to rotate, but not to tip, to best fit the electron density.

## Figures

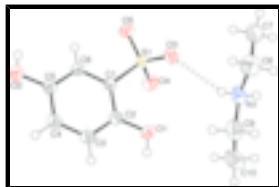


Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary sphere for the H atom). The hydrogen bond is shown as a double-dashed line.

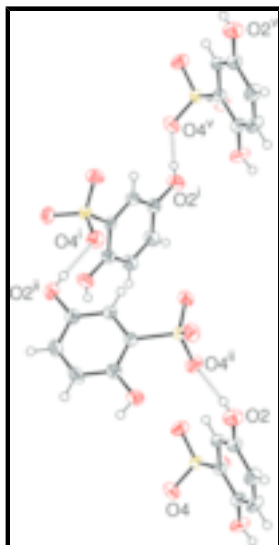


Fig. 2. Part of a helical chain on anions in the crystal structure of (I). The hydrogen bonds are shown as a double-dashed lines. Symmetry codes as in Table 1; additionally: (v)  $x, y, z + 1$ .

## Diethylammonium 2,5-dihydroxybenzenesulfonate

### Crystal data

$C_4H_{12}N^+ \cdot C_6H_5O_5S^-$

$M_r = 263.31$

Trigonal,  $P3_2$

Hall symbol: P 32

$a = 8.7006 (4) \text{ \AA}$

$b = 8.7006 (4) \text{ \AA}$

$c = 14.0732 (6) \text{ \AA}$

$\alpha = 90^\circ$

$\beta = 90^\circ$

$\gamma = 120^\circ$

$V = 922.62 (7) \text{ \AA}^3$

$Z = 3$

$F_{000} = 420$

$D_x = 1.422 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5152 reflections

$\theta = 2.7\text{--}29.9^\circ$

$\mu = 0.27 \text{ mm}^{-1}$

$T = 291 (2) \text{ K}$

Chunk, colourless

$0.40 \times 0.35 \times 0.30 \text{ mm}$

### Data collection

Bruker SMART1000 CCD  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

3516 independent reflections

3307 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

$T = 291(2)$  K  $\theta_{\max} = 30.0^\circ$   
 $\omega$  scans  $\theta_{\min} = 2.7^\circ$   
 Absorption correction: multi-scan (SADABS; Bruker, 1999)  $h = -12 \rightarrow 8$   
 $T_{\min} = 0.900$ ,  $T_{\max} = 0.926$   $k = -10 \rightarrow 12$   
 7627 measured reflections  $l = -19 \rightarrow 19$

### Refinement

Refinement on  $F^2$  Hydrogen site location: difmap and geom  
 Least-squares matrix: full H atoms treated by a mixture of independent and constrained refinement  
 $R[F^2 > 2\sigma(F^2)] = 0.029$   $w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.0286P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $wR(F^2) = 0.079$   $(\Delta/\sigma)_{\max} < 0.001$   
 $S = 1.08$   $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$   
 3516 reflections  $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$   
 169 parameters Extinction correction: SHELXL97 (Sheldrick, 1997),  
 $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 1 restraint Extinction coefficient: 0.028 (3)  
 Primary atom site location: structure-invariant direct methods Absolute structure: Flack (1983), with 1734 Friedel pairs  
 Secondary atom site location: difference Fourier map Flack parameter: 0.03 (5)

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.92539 (18)	0.24787 (18)	0.04160 (9)	0.0255 (2)
C2	0.96186 (19)	0.3460 (2)	-0.04236 (11)	0.0303 (3)
C3	1.1272 (2)	0.4984 (2)	-0.05275 (13)	0.0379 (3)
H3A	1.1536	0.5643	-0.1084	0.046*
C4	1.2525 (2)	0.5526 (2)	0.01894 (12)	0.0379 (4)
H4A	1.3625	0.6544	0.0110	0.045*
C5	1.2151 (2)	0.45559 (19)	0.10314 (11)	0.0304 (3)
C6	1.05126 (19)	0.30364 (19)	0.11403 (10)	0.0268 (3)
H6	1.0251	0.2385	0.1700	0.032*

## supplementary materials

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O1	0.83456 (17)	0.28992 (19)	-0.11154 (9)	0.0413 (3)
H1	0.879 (3)	0.367 (3)	-0.1556 (17)	0.050*
O2	1.34355 (16)	0.51443 (18)	0.17195 (9)	0.0413 (3)
H2	1.305 (4)	0.454 (4)	0.2142 (19)	0.050*
S1	0.72428 (4)	0.04463 (4)	0.05746 (2)	0.02561 (9)
O3	0.57761 (15)	0.08132 (17)	0.05299 (8)	0.0369 (3)
O4	0.71698 (18)	-0.07059 (17)	-0.02011 (9)	0.0399 (3)
O5	0.73716 (16)	-0.02270 (16)	0.14986 (8)	0.0383 (3)
C7	0.1436 (3)	-0.0537 (3)	0.03267 (17)	0.0538 (5)
H7A	0.0917	-0.0234	0.0844	0.081*
H7B	0.2264	-0.0862	0.0572	0.081*
H7C	0.0519	-0.1517	-0.0020	0.081*
C8	0.2377 (3)	0.1017 (3)	-0.03175 (17)	0.0551 (5)
H8A	0.1524	0.1297	-0.0592	0.066*
H8B	0.3221	0.2037	0.0049	0.066*
C9	0.4290 (3)	0.2214 (3)	-0.17701 (17)	0.0513 (5)
H9A	0.3551	0.2732	-0.1905	0.062*
H9B	0.5373	0.3120	-0.1473	0.062*
C10	0.4738 (3)	0.1650 (3)	-0.2678 (2)	0.0615 (6)
H10A	0.5233	0.2628	-0.3115	0.092*
H10B	0.3682	0.0682	-0.2946	0.092*
H10C	0.5588	0.1276	-0.2557	0.092*
N1	0.3328 (2)	0.0685 (2)	-0.10904 (12)	0.0427 (3)
H3	0.401 (4)	0.038 (4)	-0.0891 (18)	0.051*
H4	0.238 (4)	-0.038 (4)	-0.1357 (17)	0.051*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0235 (6)	0.0256 (6)	0.0259 (6)	0.0113 (5)	0.0015 (5)	0.0008 (5)
C2	0.0293 (7)	0.0332 (7)	0.0295 (6)	0.0165 (6)	0.0000 (5)	0.0052 (6)
C3	0.0343 (7)	0.0359 (8)	0.0384 (8)	0.0136 (6)	0.0028 (6)	0.0152 (6)
C4	0.0309 (7)	0.0303 (7)	0.0419 (8)	0.0073 (6)	-0.0002 (6)	0.0094 (6)
C5	0.0274 (7)	0.0290 (6)	0.0312 (6)	0.0113 (6)	-0.0017 (5)	0.0003 (6)
C6	0.0276 (6)	0.0274 (6)	0.0239 (6)	0.0127 (5)	-0.0003 (5)	0.0012 (5)
O1	0.0362 (6)	0.0485 (7)	0.0302 (5)	0.0144 (5)	-0.0053 (4)	0.0098 (5)
O2	0.0308 (6)	0.0401 (6)	0.0370 (6)	0.0058 (5)	-0.0081 (5)	-0.0009 (5)
S1	0.02316 (15)	0.02566 (15)	0.02480 (14)	0.00979 (12)	0.00046 (11)	-0.00058 (12)
O3	0.0266 (5)	0.0431 (6)	0.0429 (6)	0.0190 (5)	0.0036 (4)	0.0021 (5)
O4	0.0428 (6)	0.0370 (6)	0.0393 (6)	0.0195 (5)	-0.0030 (5)	-0.0125 (5)
O5	0.0373 (6)	0.0349 (6)	0.0320 (5)	0.0100 (5)	-0.0004 (4)	0.0096 (4)
C7	0.0437 (10)	0.0623 (13)	0.0535 (11)	0.0251 (9)	-0.0015 (9)	-0.0083 (9)
C8	0.0630 (13)	0.0535 (11)	0.0598 (12)	0.0373 (11)	-0.0016 (10)	-0.0107 (9)
C9	0.0430 (10)	0.0388 (9)	0.0690 (13)	0.0180 (8)	-0.0050 (9)	0.0055 (9)
C10	0.0509 (12)	0.0484 (11)	0.0776 (15)	0.0190 (10)	0.0137 (11)	0.0141 (11)
N1	0.0336 (7)	0.0376 (7)	0.0604 (10)	0.0204 (6)	0.0008 (7)	-0.0020 (7)

*Geometric parameters (Å, °)*

C1—C6	1.3937 (19)	C7—C8	1.488 (4)
C1—C2	1.3982 (19)	C7—H7A	0.9600
C1—S1	1.7732 (14)	C7—H7B	0.9600
C2—O1	1.3682 (18)	C7—H7C	0.9600
C2—C3	1.393 (2)	C8—N1	1.480 (3)
C3—C4	1.384 (2)	C8—H8A	0.9700
C3—H3A	0.9300	C8—H8B	0.9700
C4—C5	1.396 (2)	C9—C10	1.489 (4)
C4—H4A	0.9300	C9—N1	1.507 (3)
C5—O2	1.370 (2)	C9—H9A	0.9700
C5—C6	1.385 (2)	C9—H9B	0.9700
C6—H6	0.9300	C10—H10A	0.9600
O1—H1	0.85 (2)	C10—H10B	0.9600
O2—H2	0.75 (3)	C10—H10C	0.9600
S1—O5	1.4533 (12)	N1—H3	0.82 (3)
S1—O4	1.4618 (12)	N1—H4	0.96 (3)
S1—O3	1.4635 (13)		
C6—C1—C2	120.44 (13)	H7A—C7—H7B	109.5
C6—C1—S1	117.84 (10)	C8—C7—H7C	109.5
C2—C1—S1	121.65 (11)	H7A—C7—H7C	109.5
O1—C2—C3	122.09 (14)	H7B—C7—H7C	109.5
O1—C2—C1	119.18 (13)	N1—C8—C7	112.22 (18)
C3—C2—C1	118.74 (13)	N1—C8—H8A	109.2
C4—C3—C2	120.70 (14)	C7—C8—H8A	109.2
C4—C3—H3A	119.6	N1—C8—H8B	109.2
C2—C3—H3A	119.6	C7—C8—H8B	109.2
C3—C4—C5	120.46 (14)	H8A—C8—H8B	107.9
C3—C4—H4A	119.8	C10—C9—N1	112.02 (17)
C5—C4—H4A	119.8	C10—C9—H9A	109.2
O2—C5—C6	122.44 (14)	N1—C9—H9A	109.2
O2—C5—C4	118.29 (13)	C10—C9—H9B	109.2
C6—C5—C4	119.27 (14)	N1—C9—H9B	109.2
C5—C6—C1	120.39 (13)	H9A—C9—H9B	107.9
C5—C6—H6	119.8	C9—C10—H10A	109.5
C1—C6—H6	119.8	C9—C10—H10B	109.5
C2—O1—H1	105.5 (17)	H10A—C10—H10B	109.5
C5—O2—H2	108 (2)	C9—C10—H10C	109.5
O5—S1—O4	112.17 (8)	H10A—C10—H10C	109.5
O5—S1—O3	112.19 (7)	H10B—C10—H10C	109.5
O4—S1—O3	111.96 (7)	C8—N1—C9	114.35 (16)
O5—S1—C1	106.14 (7)	C8—N1—H3	112.5 (18)
O4—S1—C1	105.83 (7)	C9—N1—H3	109.5 (19)
O3—S1—C1	108.08 (7)	C8—N1—H4	100.4 (15)
C8—C7—H7A	109.5	C9—N1—H4	115.9 (15)
C8—C7—H7B	109.5	H3—N1—H4	104 (2)
C6—C1—C2—O1	-178.78 (14)	C2—C1—C6—C5	-1.0 (2)

## supplementary materials

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S1—C1—C2—O1	4.4 (2)	S1—C1—C6—C5	176.03 (12)
C6—C1—C2—C3	1.1 (2)	C6—C1—S1—O5	0.19 (13)
S1—C1—C2—C3	-175.75 (13)	C2—C1—S1—O5	177.14 (12)
O1—C2—C3—C4	179.34 (17)	C6—C1—S1—O4	-119.17 (12)
C1—C2—C3—C4	-0.6 (3)	C2—C1—S1—O4	57.78 (13)
C2—C3—C4—C5	-0.2 (3)	C6—C1—S1—O3	120.73 (11)
C3—C4—C5—O2	179.88 (16)	C2—C1—S1—O3	-62.32 (13)
C3—C4—C5—C6	0.4 (3)	C7—C8—N1—C9	179.41 (18)
O2—C5—C6—C1	-179.29 (14)	C10—C9—N1—C8	-162.32 (19)
C4—C5—C6—C1	0.2 (2)		

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ O3 <sup>i</sup>	0.85 (2)	2.02 (3)	2.8441 (17)	163 (2)
O2—H2 $\cdots$ O4 <sup>ii</sup>	0.75 (3)	1.90 (3)	2.6480 (19)	174 (3)
N1—H3 $\cdots$ O3	0.82 (3)	2.43 (3)	3.084 (2)	138 (2)
N1—H3 $\cdots$ O2 <sup>iii</sup>	0.82 (3)	2.49 (3)	3.0691 (19)	129 (2)
N1—H4 $\cdots$ O5 <sup>iv</sup>	0.96 (3)	1.94 (3)	2.888 (2)	171 (2)

Symmetry codes: (i)  $-y+1, x-y, z-1/3$ ; (ii)  $-x+y+2, -x+1, z+1/3$ ; (iii)  $-y+1, x-y-1, z-1/3$ ; (iv)  $-y, x-y-1, z-1/3$ .



Fig. 1

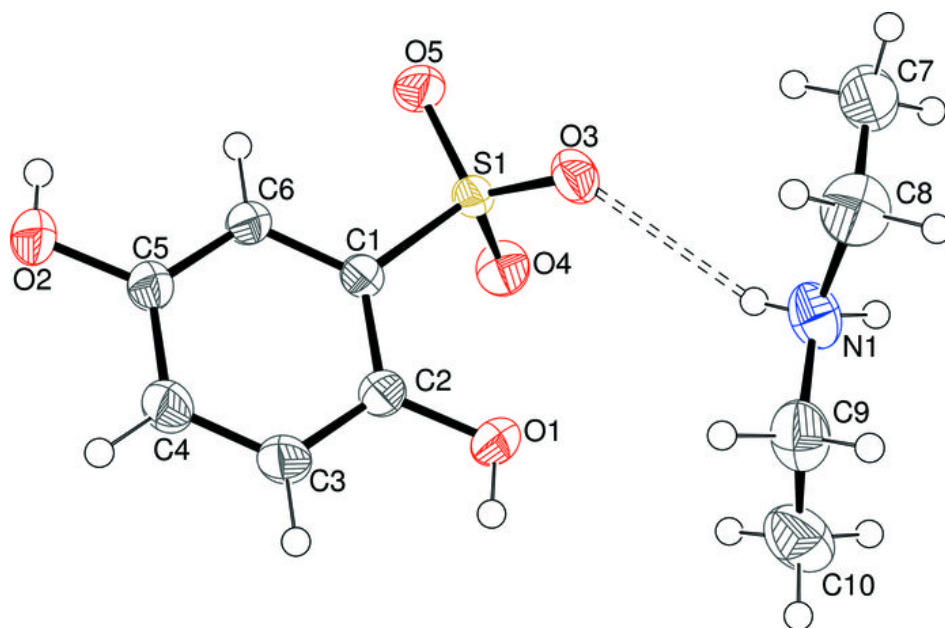


Fig. 2

