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Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.009 \text{ \AA}$

R factor = 0.053

wR factor = 0.052

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>.2-Hydroxy-3-(*p*-nitrobenzenesulfonylamino)propyl-
ammonium *p*-nitrobenzenesulfonate

The title compound, $[\text{NO}_2\text{C}_6\text{H}_4\text{SO}_2\text{NHCH}_2\text{CHOHCH}_2\text{NH}_3]^+ [p\text{-NO}_2\text{C}_6\text{H}_4\text{SO}_3]^-$ or $\text{C}_9\text{H}_{14}\text{N}_3\text{O}_5\text{S}^+ \cdot \text{C}_6\text{H}_4\text{NO}_5\text{S}^-$, was isolated from the reaction between 1,3-diamino-2-hydroxypropane and *p*-nitrobenzenesulfonyl chloride in CH_2Cl_2 . The fairly extensive hydrogen-bond system involving all 'active' H atoms links the ions in the crystal into layers normal to the *b* axis.

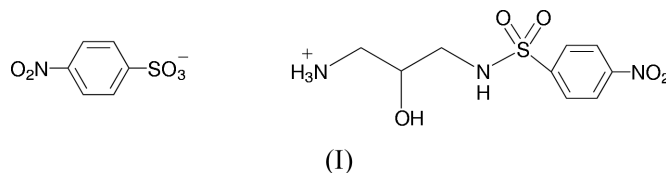
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Comment

Nitrobenzenesulfonamide is an efficient protecting reagent for preparing amine derivatives (Fukuyama *et al.*, 1995; Hidai *et al.*, 1999). Under controlled reaction conditions, a mono-protected primary diamine was synthesized selectively and in high yield. The crystal of the title compound, (I), has an ionic structure, built of 2-hydroxy-3-(*p*-nitrobenzenesulfonylamino)propylammonium cations, $[\text{NO}_2\text{C}_6\text{H}_4\text{SO}_2\text{NHCH}_2\text{CHOHCH}_2\text{NH}_3]^+$, and *p*-nitrobenzenesulfonate anions, $[p\text{-NO}_2\text{-C}_6\text{H}_4\text{SO}_3]^-$, as shown in Fig. 1. The present X-ray diffraction study proved the successful formation of mono-substituted products and provided the first reported crystal structure with cationic mono-protected diamine.



The S—O and S—N bond lengths in the cation, and the S—O and S—C bond lengths in the anion are comparable to those in similar previously reported molecular fragments (Barrans & Cotrait, 1976; Brunzelle *et al.*, 1999; Cook *et al.*, 1971; Curtis & Pavkovic, 1983; Russell *et al.*, 1994; Takahashi *et al.*, 1998).

There are five 'active' H atoms in the structure of (I), all of which participate in hydrogen bonds of the N—H...O=S, N—H...O(H) and O—H...O=S types (Table 2). The N—H...O=S and N—H...O(H) bonds are in the range 2.809 (5)–3.038 (5) Å. O5—H5A...O10 is a fairly strong interaction, with an O...O distance of 2.791 (5) Å and an O—H...O angle of 164.85 (18)°, which is typical for 'anionic' O—H...O[−] hydrogen bonds in the crystal structures of salts (Jeffrey, 1997). These hydrogen bonds link cations and anions into infinite two-dimensional aggregates, *i.e.* layers parallel to the (010) plane (Fig. 2).

Experimental

1,3-Diamino-2-hydroxypropane (0.2 g) was stirred with *p*-nitrobenzenesulfonyl chloride (0.5 g) and pyridine in dry CH_2Cl_2 (10 ml) under N_2 at room temperature for 5 h. The reaction mixture was extracted with 5% aqueous HCl solution. The organic phase was

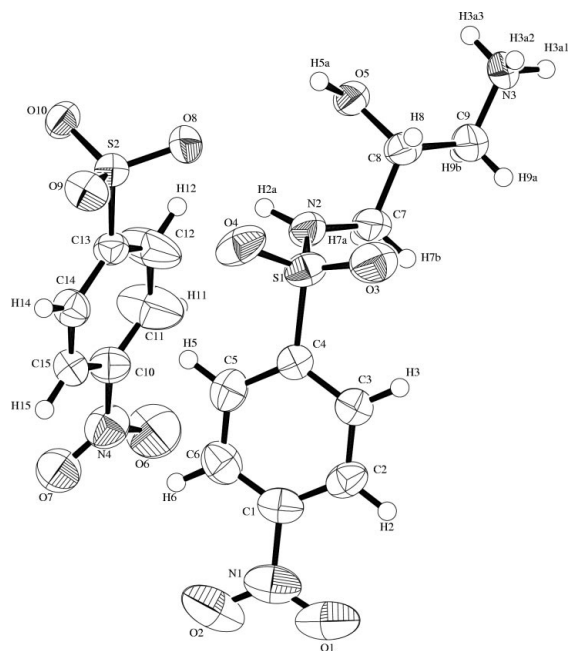


Figure 1
An ORTEP (Johnson, 1976) drawing of the cation and anion in the structure of (I), showing 50% probability displacement ellipsoids and the crystallographic labelling scheme.

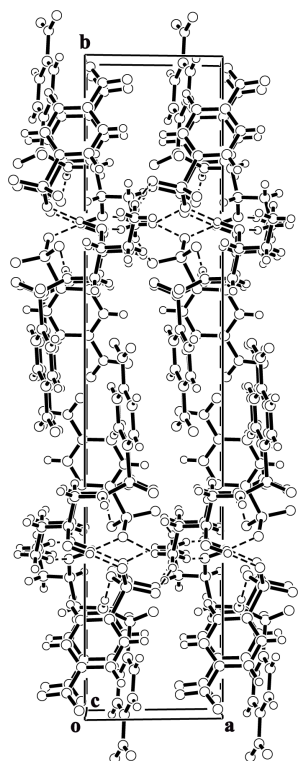


Figure 2
Packing diagram, showing the hydrogen-bonding system.

dried and purified by silica chromatography [elution with CH_2Cl_2 -MeOH, 50:1 (v:v)]. The pale-yellow monosulfonylated diamine was obtained in 75% yield. X-ray quality crystals were obtained by slow

evaporation of a 10 ml solution in CH_2Cl_2 and MeOH in a 3:1 (v:v) ratio at room temperature over a few days.

Crystal data

$\text{C}_9\text{H}_{14}\text{N}_3\text{O}_5\text{S}^+\cdot\text{C}_6\text{H}_4\text{NO}_5\text{S}^-$
 $M_r = 478.45$
 Monoclinic, $P2_1/c$
 $a = 6.7120$ (8) Å
 $b = 31.392$ (1) Å
 $c = 9.8260$ (9) Å
 $\beta = 103.170$ (5)°
 $V = 2015.9$ (3) Å³
 $Z = 4$

$D_x = 1.576$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 46 reflections
 $\theta = 2.5$ – 14°
 $\mu = 0.33$ mm⁻¹
 $T = 298$ K
 Block, colourless
 $0.23 \times 0.20 \times 0.11$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 12 619 measured reflections
 4629 independent reflections
 1992 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.049$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -8 \rightarrow 5$
 $k = -38 \rightarrow 40$
 $l = -8 \rightarrow 12$

Refinement

Refinement on F^2
 $R = 0.053$
 $wR = 0.052$
 $S = 1.19$
 1992 reflections
 280 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o) + (PF_o)^2]$
 where $P = 0.0205$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

S1—O3	1.421 (4)	S2—C13	1.770 (5)
S1—O4	1.436 (4)	O5—C8	1.434 (6)
S1—N2	1.594 (4)	N2—C7	1.457 (6)
S1—C4	1.767 (5)	N3—C9	1.481 (6)
S2—O8	1.452 (3)	C7—C8	1.535 (7)
S2—O9	1.444 (3)	C8—C9	1.468 (7)
S2—O10	1.459 (3)		
O3—S1—O4	120.4 (2)	S1—C4—C3	119.1 (4)
O3—S1—N2	110.0 (2)	S1—C4—C5	121.2 (4)
O3—S1—C4	105.9 (2)	N2—C7—C8	109.8 (4)
O4—S1—N2	106.1 (2)	O5—C8—C7	109.0 (4)
O4—S1—C4	107.3 (2)	O5—C8—C9	109.3 (4)
N2—S1—C4	106.3 (2)	C7—C8—C9	111.3 (4)
S1—N2—C7	123.3 (4)	N3—C9—C8	112.7 (4)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A ⁱ ···O8	0.95	1.92	2.852 (5)	166
N3—H3A1 ⁱ ···O10 ⁱ	0.95	1.91	2.809 (5)	157
N3—H3A2 ⁱ ···O5 ⁱⁱ	0.95	2.12	3.002 (5)	154
N3—H3A3 ⁱ ···O5	0.95	2.55	2.871 (5)	100
N3—H3A3 ⁱ ···O9 ⁱⁱⁱ	0.95	2.14	3.038 (5)	158
O5—H5A ⁱ ···O10 ⁱⁱ	1.11	1.70	2.791 (5)	165

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

The positions of the H atoms were generated geometrically (C—H and N—H bond lengths fixed at 0.95 Å), with assigned isotropic displacement parameters, and were made to ride on their respective parent C and N atoms before the final cycle of least-squares refinement.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT-Plus (Bruker, 1999); program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to

refine structure: *TEXSAN* (Molecular Structure Corporation, 1992); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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