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Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.032
 wR factor = 0.034
Data-to-parameter ratio = 18.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Iminodiethylenediaminium bis(2,5-dichloro-
benzenesulfonate)

In the title compound, $\text{C}_4\text{H}_{15}\text{N}_3^{2+} \cdot 2\text{C}_6\text{H}_3\text{Cl}_2\text{O}_3\text{S}^-$, the bis(2-aminoethyl)amine is protonated at the N atoms of both aminoethyl groups. Crystal packing is stabilized by a network of strong $\text{N}-\text{H} \cdots \text{O}$ hydrogen-bond interactions involving all the ions.

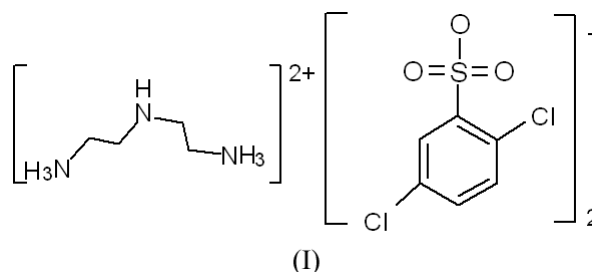
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Comment

Organophilic clays are widely used for their rheological properties in industrial applications, such as cosmetics, inks and paints (Jones, 1983). New developments in the application of organo-exchanged layered silicates have been reviewed by Ogawa & Kuroda (1997). Various types of organic cations have been used to prepare organo-clays. These include several kinds of organic ammonium cations (TMA^+) and their long-chain alkyltrimethylammonium homologues, the trimethylphenylammonium cation (TMPA^+) and alkyipyridinium cations. Recently, our work has focused on the synthesis of uncommon organic ammonium salts, and their application to modifying sodium montmorillonite. We report here the crystal structure of the title compound, (I), selected from a series of synthesized organic ammonium salts.



The asymmetric unit of (I) contains one bis(2-aminoethyl)amine cation and two 2,5-dichlorobenzenesulfonate anions. Bis(2-aminoethyl)amine is protonated at the N atoms of the aminoethyl groups. The mean $\text{S}-\text{O}$ distance of 1.449 Å observed in this structure is coincident with the average value of 1.45 Å found in the structures of 2,5-dibromo- and 2,5-dichlorobenzenesulfonic acids (Lundgren & Lundin, 1972). The small but significant differences observed for the $\text{S}-\text{O}$ bond distances could indicate that the delocalization of the negative charge on the SO_3^- group is affected by the Cl substituents on the benzene ring. The crystal packing is characterized by the presence of strong $\text{N}-\text{H} \cdots \text{O}$ intermolecular hydrogen-bond interactions, as detailed in Table 2 and Fig. 2.

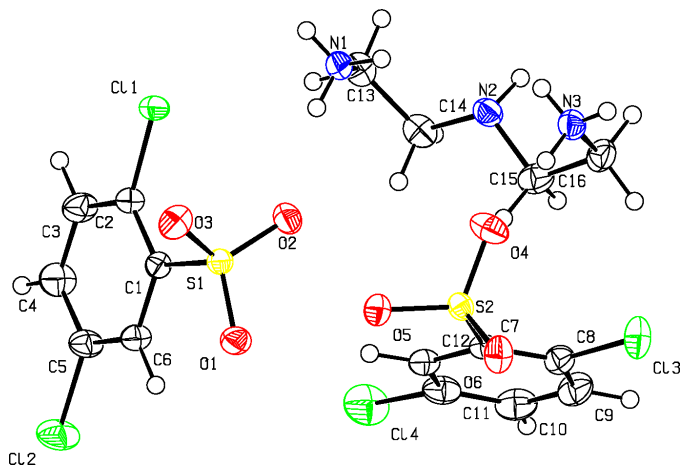


Figure 1
The asymmetric unit of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

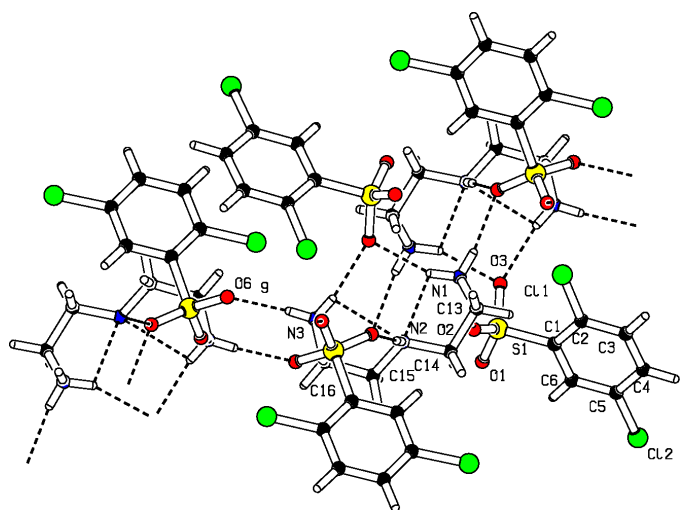


Figure 2
The packing of the title compound, showing intermolecular hydrogen bonds as dashed lines.

Experimental

The title compound was prepared by mixing 2,5-dichlorobenzene-sulfonic acid (0.526 g, 2.00 mmol) dissolved in methanol (10 ml) and bis(2-aminoethyl)amine (0.103 g, 1.00 mmol) in methanol (5 ml). The resulting solution was kept in the dark at room temperature. Colourless crystals separated from the solution after *ca* one week. Analysis calculated for $C_{16}H_{21}Cl_4N_3O_6S_2$: C 34.48, H 3.80, N 7.54%; found: C 34.39, H 3.81, N 7.58%.

Crystal data

$C_{16}H_{21}N_3^{2+} \cdot 2C_6H_3Cl_2O_3S^-$
 $M_r = 557.29$
 Monoclinic, $P2_1/n$
 $a = 12.924 (4) \text{ \AA}$
 $b = 8.644 (3) \text{ \AA}$
 $c = 21.250 (5) \text{ \AA}$
 $\beta = 103.25 (2)^\circ$
 $V = 2310.8 (13) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.602 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 19 169 reflections
 $\theta = 3.0\text{--}27.5^\circ$
 $\mu = 0.73 \text{ mm}^{-1}$
 $T = 173.1 \text{ K}$
 Cube, colourless
 $0.30 \times 0.30 \times 0.30 \text{ mm}$

Data collection

Rigaku R-Axis RAPID diffractometer
 ω scans
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.776, T_{\max} = 0.803$
 22 603 measured reflections

5288 independent reflections
 4061 reflections with $I > 3\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 27.5^\circ$
 $h = -16 \rightarrow 14$
 $k = -11 \rightarrow 11$
 $l = -27 \rightarrow 27$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.034$
 $S = 1.03$
 5288 reflections
 280 parameters
 H-atom parameters constrained
 $w = [1 - (F_o - F_c)^2/6\sigma^2(F)]^2 / [3.30T_0(x) - 0.458T_1(x) + 2.17T_2(x)]$
 where T_i are Chebyshev polynomials and $x = F_o/F_{\max}$ (Watkin, 1994; Prince, 1982)

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.47 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

C1—C2	1.7371 (18)	S2—O4	1.4468 (15)
C12—C5	1.7395 (15)	S2—O5	1.4512 (13)
C13—C8	1.7349 (19)	S2—O6	1.4513 (13)
C14—C11	1.742 (2)	S2—C7	1.7853 (18)
S1—O1	1.4392 (14)	N1—C13	1.486 (2)
S1—O2	1.4475 (14)	N2—C14	1.462 (2)
S1—O3	1.4589 (14)	N2—C15	1.455 (2)
S1—C1	1.7891 (15)	N3—C16	1.480 (2)
O1—S1—C1	105.34 (8)	O5—S2—C7	105.59 (8)
O1—S1—O2	113.78 (8)	O6—S2—C7	106.25 (8)
O1—S1—O3	112.67 (8)	C14—N2—C15	113.93 (15)
O3—S1—C1	105.29 (8)	N1—C13—C14	108.70 (14)
O2—S1—O3	112.39 (8)	N2—C14—C13	109.46 (16)
O2—S1—C1	106.53 (8)	N2—C15—C16	110.53 (15)
O4—S2—O5	112.59 (8)	N3—C16—C15	111.27 (15)
O5—S2—O6	112.50 (8)	S2—C7—C8	122.35 (13)
O4—S2—O6	113.06 (8)	S2—C7—C12	118.13 (13)
O4—S2—C7	106.13 (8)		

Table 2

Hydrogen-bonding geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H7 \cdots O3 ⁱ	0.89	2.08	2.9242 (19)	158
N1—H8 \cdots O5 ⁱ	0.84	2.02	2.8475 (19)	167
N1—H9 \cdots O2	0.88	1.99	2.830 (2)	160
N2—H14 \cdots O5 ⁱⁱ	0.94	2.18	3.1163 (19)	170
N3—H19 \cdots O3 ⁱ	0.84	2.08	2.854 (2)	155
N3—H20 \cdots O6 ⁱⁱⁱ	0.81	2.02	2.823 (2)	168
N3—H21 \cdots O4	0.90	1.90	2.799 (2)	174

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

H atoms were positioned geometrically after each cycle and allowed to ride on their parent atoms; N—H = 0.81–0.94 \AA and C—H = 1.00 \AA , with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 0.05 \text{ \AA}^2$ for those bonded to N atoms.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *CrystalStructure*.

References

- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, C. K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
- Lundgren, J. O. & Lundin, P. (1972). *Acta Cryst.* **B28**, 486–491.
- Jones, T. R. (1983). *Clay Miner.* **18**, 399–410.
- Ogawa, M. & Kuroda, K. (1997). *Bull. Chem. Soc. Jpn.*, **70**, 2593–2618.
- Prince, E. (1982). *Mathematical Techniques in Crystallography and Materials Science*. New York: Springer-Verlag.
- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MSK (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MSK, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Watkin, D. J. (1994). *Acta Cryst.* **A50**, 411–437.