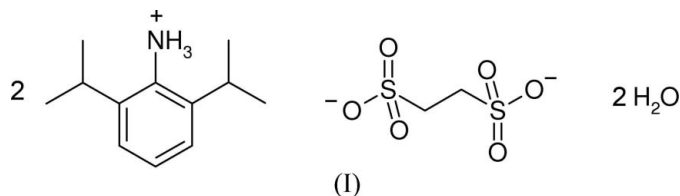


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Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.041
 wR factor = 0.108
Data-to-parameter ratio = 14.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis[(2,6-diisopropylphenyl)ammonium]
ethane-1,2-disulfonate dihydrateThe title compound, $2\text{C}_{12}\text{H}_{20}\text{N}^+ \cdot \text{C}_2\text{H}_4\text{O}_6\text{S}_2^{2-} \cdot 2\text{H}_2\text{O}$, crystallizes with the edisylate anion situated on a center of inversion. All conventional hydrogen-bond donors and acceptors are utilized, forming two-dimensional sheets.Received 15 May 2006
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Comment

(2,6-Diisopropylphenyl)amine (DIPA), is frequently used as a starting material for pharmaceutical synthesis. Ethane-1,2-disulfonic acid (edisylate acid) is a desirable acid for salt formation; its difunctionality, large $\text{p}K_a$ and flexibility can accommodate hydrogen-bond donors very well. During efforts to purify DIPA by salt formation and crystallization, we formed the title 2:1 salt (Fig. 1).

Numerous attempts were made to synthesize the 1:1 salt, in which the edisylate anion would be singly deprotonated. Even though the acid/base pairs appear appropriate for such a compound to form, it could not be obtained from over 25 crystallization experiments, conducted with a variety of conditions and solvents. The 2:1 salt has a low critical water activity; we observed the hydrate forming out of chloroform and other non-hygroscopic organic solvents. In the crystal structure, the water molecule donates both protons and accepts a proton as part of the hydrogen bonding. This is the most common environment for an organic hydrate (Gillon *et al.*, 2003).

The structure has all conventional hydrogen-bond donors and acceptors used. The protonated NH_3 groups face the counter-ion and water molecule for hydrogen bonding, forming two-dimensional sheets parallel to the (001) planes. The DIPAH^+ cations interleave between sheets with hydrophobic ends facing each other. While several intermolecular contacts from the polar end of DIPAH^+ are within the sum of the relevant van der Waals radii, there are no such contacts at the hydrophobic ends.

The torsion angles from the aromatic group to the isopropyl groups are interesting. The expectation is that the isopropyl groups would eclipse the aromatic ring plane so that the C5—H5 and C8—H8 bonds lie in the same plane as atoms N1, C3, C4 and C6. One of these groups exhibits such a conformation

(C3—C6—C5—H5 = 3°), while the other shows a slight twist (C3—C4—C8—H8 = 36°). The C5—H5 bond points between H99B and H99C bonded to N1, while the C8—H8 bond points towards H99A. An interaction between H8 and H99A may be responsible for the twist of the C8—H8 bond out of the ring plane. This same feature is present in another structure with DIPA (Bond & Doyle, 2003).

Experimental

A stock solution of DIPA was made in methanol (25 mg ml⁻¹). To a crystallizer vessel, 0.12 ml of stock solution was added with 0.5 equivalents of ethane-1,2-disulfonic acid (50 mM in water). The sample was evaporated to dryness, dissolved in chloroform and allowed to evaporate over 24 h. A crystal of (I) was removed from the crystallizer plate and mounted on a mitogen loop with paratone-N oil.

Crystal data

2C ₁₂ H ₂₀ N ⁺ ·C ₂ H ₄ O ₆ S ₂ ²⁻ ·2H ₂ O	V = 786.6 (5) Å ³
M _r = 580.78	Z = 1
Triclinic, P $\bar{1}$	D _x = 1.226 Mg m ⁻³
a = 6.0827 (19) Å	Mo K α radiation
b = 9.422 (3) Å	μ = 0.22 mm ⁻¹
c = 13.909 (5) Å	T = 173 (2) K
α = 81.476 (5)°	Plate, colorless
β = 89.700 (5)°	0.23 × 0.21 × 0.05 mm
γ = 86.211 (5)°	

Data collection

Bruker SMART APEX CCD diffractometer	11376 measured reflections
φ and ω scans	2883 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	2557 reflections with $I > 2\sigma(I)$
T _{min} = 0.877, T _{max} = 0.990	R _{int} = 0.026
	θ_{max} = 25.4°

Refinement

Refinement on F ²	$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 0.366P]$
R[F ² > 2 σ (F ²)] = 0.041	where $P = (F_o^2 + 2F_c^2)/3$
wR(F ²) = 0.108	(Δ/σ) _{max} < 0.001
S = 1.04	$\Delta\rho_{max} = 0.39 \text{ e } \text{Å}^{-3}$
2883 reflections	$\Delta\rho_{min} = -0.24 \text{ e } \text{Å}^{-3}$
196 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H98A...O4 ⁱ	0.83 (3)	1.99 (3)	2.814 (2)	175 (3)
O1—H98B...O5	0.78 (3)	2.01 (3)	2.777 (2)	170 (3)
N1—H99C...O1 ⁱⁱ	0.93 (2)	1.78 (2)	2.706 (2)	178 (2)
N1—H99B...O3 ⁱⁱⁱ	0.91 (2)	1.83 (2)	2.722 (2)	170 (2)
N1—H99A...O4 ^{iv}	0.88 (3)	2.04 (2)	2.842 (2)	150 (2)

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

H atoms bound to C atoms were placed in idealized positions and refined using a riding model with C—H = 0.95 Å for Csp², 0.98 Å for CH₃, 0.99 Å for CH₂ and 1.00 Å for CH. U_{iso}(H) values were set at 1.5U_{eq}(C) for the methyl groups and 1.2U_{eq}(C) in all other cases. H atoms bound to N or O atoms were located in difference Fourier maps and their positions were refined freely with isotropic displace-

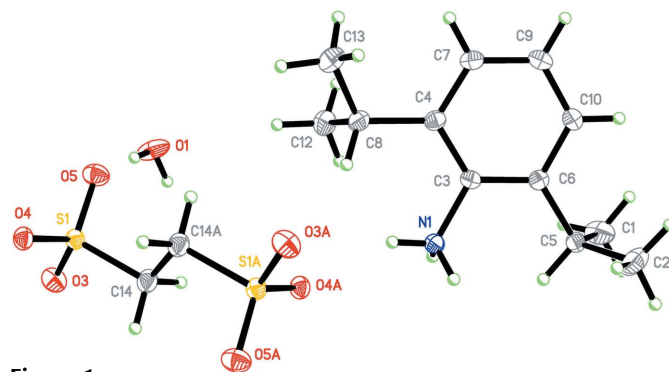


Figure 1

View of the constituents of (I), showing the atom-labeling scheme and displacement ellipsoids drawn at the 30% probability level. H atoms are represented by circles of arbitrary size. [Symmetry code: (A) $-x + 2, -y + 1, -z + 1$].

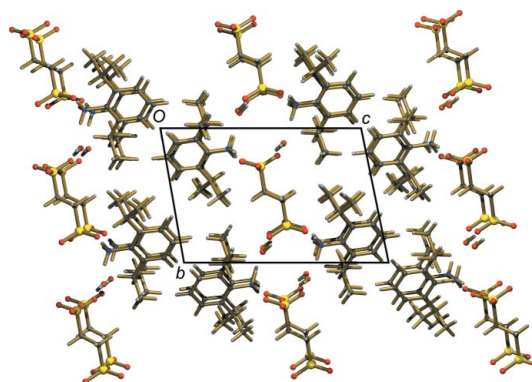


Figure 2

The structure viewed along the *a* direction, showing two-dimensional hydrogen-bonded sheets parallel to the (001) planes, with the DIPA⁺ cations interleaved between them.

ment parameters. Refined N—H and O—H distances are included in Table 1.

Data collection: SMART (Bruker, 2003); cell refinement: SMART; data reduction: SAINT-Plus (Bruker, 2003); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2003), PLATON (Spek, 2003) and POV-RAY (Persistence of Vision, 2004); software used to prepare material for publication: SHELXTL.

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