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Anion–anion dimerization in tetrabutylammonium hydrogensulfate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.007 Å
R factor = 0.073
wR factor = 0.237
Data-to-parameter ratio = 17.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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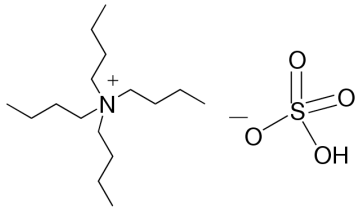
The hydrogensulfate ions of the title compound, $C_{16}H_{36}N^+\cdot HSO_4^-$, form hydrogen-bonded dimers.

Comment

The development of receptors and sensors for anions is a rapidly developing area in supramolecular chemistry (Atwood *et al.*, 1996; Beer & Gale, 2001; Beer & Smith, 1997; Bianchi *et al.*, 1997; Davis *et al.*, 1996; Kavallieratos *et al.*, 1997; Schmidtchen & Berger, 1997; Sessler & Allen, 1999). In addition, anions have recently been exploited as templates for the formation of self-assembled supramolecular architectures in both organic and inorganic systems (Gale, 2000, 2001). By drawing an analogy to carboxylic acid structures in the solid state (Bruno & Randaccio, 1980), one might expect the hydrogensulfate anions to dimerize *via* the formation of two hydrogen bonds. This anion dimer assembly has been observed in the tetramethylammonium salt (Malchus & Jansen, 1998) as well as in other crystal structures (*e.g.* Toma *et al.*, 1994), but not in the tetrabutylammonium salts that are commonly used in anion complexation studies.



(I)

The title compound, (I), crystallizes in a large monoclinic cell with four tetrabutylammonium ions and four hydrogensulfate ions in the asymmetric unit. Each hydrogensulfate ion donates and accepts a hydrogen bond (Fig. 2) to form the two dimers present in the asymmetric unit.

Experimental

The title compound was obtained from the Aldrich Chemical Company.

Crystal data

$C_{16}H_{36}N^+\cdot HSO_4^-$
M_r = 339.53
Monoclinic, $P2_1/c$
a = 21.7299 (4) Å
b = 21.3998 (4) Å
c = 17.0880 (4) Å
β = 91.318 (3)°
V = 7944.1 (3) Å^3
Z = 16
D_x = 1.136 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 38718 reflections
θ = 2.9–25.0°
μ = 0.18 mm^{-1}
T = 120 (2) K
Block, colourless
0.10 × 0.10 × 0.10 mm

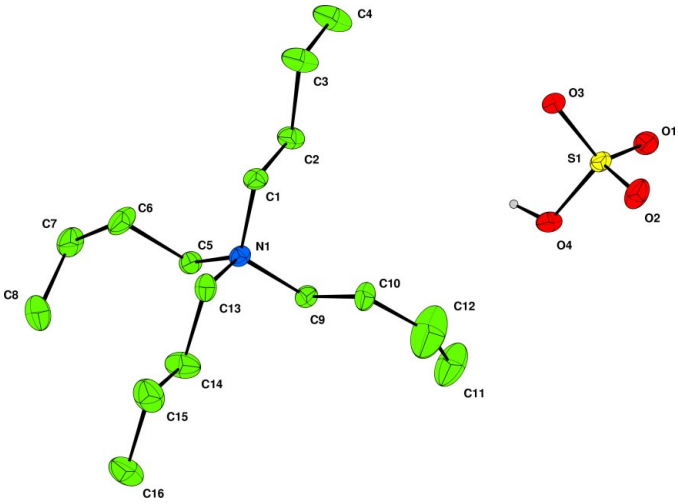


Figure 1
Displacement ellipsoid plot of one of the anion–cation pairs. The ellipsoids are drawn at the 30% probablity level and the H atoms on the tetrabutylammonium cation have been omitted for clarity.

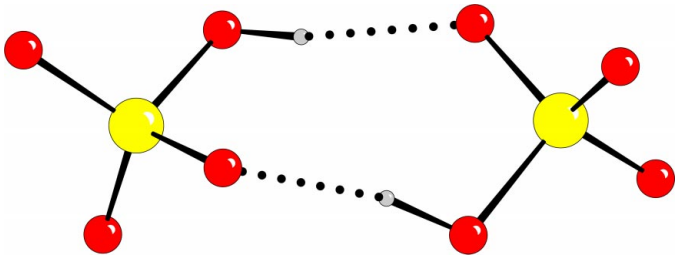


Figure 2
One of the two hydrogen-bonded hydrogensulfate dimers in the asymmetric unit..

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans to fill the Ewald sphere
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.982, T_{max} = 0.982
38 718 measured reflections
13 981 independent reflections
8004 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 25.0°
$h = -25 \rightarrow 25$
$k = -25 \rightarrow 24$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.237
S = 1.02
13 981 reflections
809 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1368P)^2 + 1.0999P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O10—H10···O3	0.79 (4)	1.85 (4)	2.628 (4)	171 (4)
O4—H4···O9	0.84 (6)	1.82 (6)	2.649 (5)	171 (6)
O8—H8···O13	0.92 (7)	1.70 (7)	2.602 (4)	166 (7)
O15—H15···O6	0.82 (8)	1.78 (8)	2.590 (5)	170 (9)

There is some thermal disorder in the ends of the tetrabutylammonium groups, but it was not found possible to model it with split sites. This results in several long and short C—C bonds. The hydroxyl H atoms were refined isotropically.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *SCALEPACK* (Otwinoski & Minor, 1997) and *COLLECT*; data reduction: *DENZO*, *COLLECT* and *maXus* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1998).

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