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N,N',N''-Triphenylguanidinium hydrogensulfate

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The hydrogensulfate anions of the title compound, $C_{19}H_{18}N_3^+ \cdot HSO_4^-$, form centrosymmetric hydrogen-bonded dimers. In the cation, the dihedral angles between the phenyl rings and the plane defined by the central guanidinium fragment are in the range 41.46 (6)–67.11 (5)°.

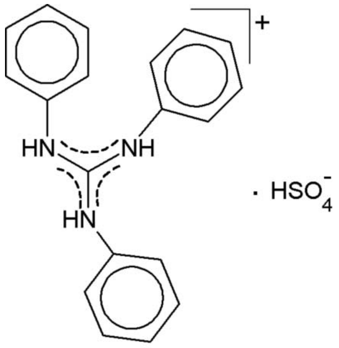
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.103
Data-to-parameter ratio = 18.0

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

Comment

The structure determination of the title compound, (I) (Fig. 1), was undertaken as part of a current research project to study the structure and physical properties (optical and dielectric) of a series of triphenylguanidine compounds.



(I)

The CN_3 fragment of the guanidinium group in (I) is planar, as expected for sp^2-hybridization of the central C atom. The bond lengths C1—N1 [1.3408 (17) Å], C1—N2 [1.3336 (18) Å] and C1—N3 [1.3329 (18) Å] are within the range expected for a delocalized C$\overset{\cdots}{-}$N bond.

The dihedral angles between the ring planes and the plane defined by the central guanidinium fragment are 54.24 (6) (C2–C7), 41.46 (6) (C8–C13) and 67.11 (5)° (C14–C19). The corresponding angles for other triphenylguanidinium salts reported in the literature are within the range 32.6 (3)–70.2 (3)° (Kemme *et al.*, 1988; Klement *et al.*, 1995; Pereira Silva *et al.*, 2006, 2007). This variability attests to the flexibility of the triphenylguanidinium cation.

In the anion, the S—O distances lie in the range 1.4420 (11)–1.5596 (11) Å, while the O—S—O angles lie between 104.60 (7) and 114.07 (7)°, indicating a distorted tetrahedral environment around the S atom. The S—O bond lengths and O—S—O bond angles of the sulfate group are consistent with the fact that only O1 has an H atom attached.

The hydrogensulfate anions donate and accept a hydrogen atom, forming centrosymmetric dimers. There are also hydrogen bonds between the NH groups and the O atoms,

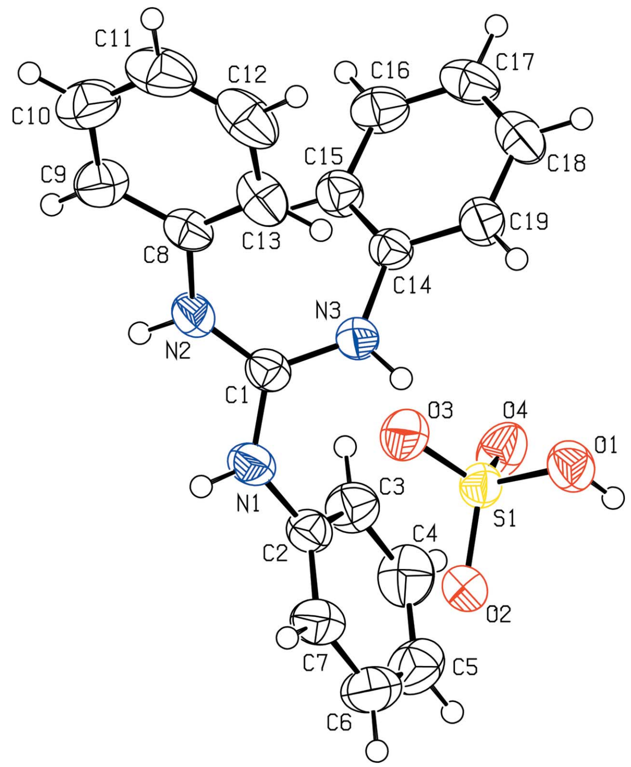


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

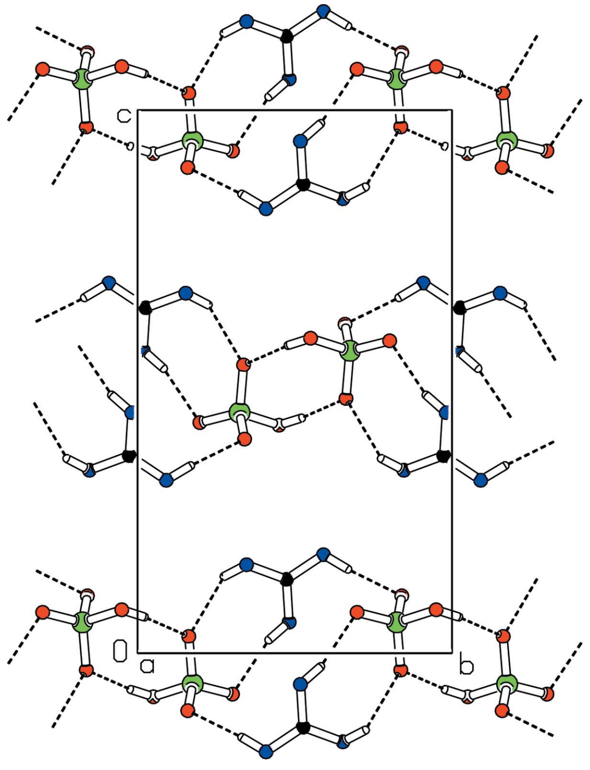


Figure 2
Packing diagram, viewed down the *b* axis, with the hydrogen bonds depicted by dashed lines. The phenyl rings have been omitted for clarity.

each anion being linked to two cations, forming infinite chains along the [010] direction (Fig. 2, Table 1). The N3—H3*A*···O4 interaction is quite strong (Table 1).

Experimental

Lithium sulfate monohydrate (Aldrich, 99%, 0.07 mmol) was added to triphenylguanidine (Aldrich, 0.07 mmol) in a water/ethanol (1:1) solution (40 ml). The solution was slowly warmed and then left to evaporate at room temperature and pressure. After a few days, small single crystals, transparent and colourless, were obtained.

Crystal data

$C_{19}H_{18}N_3^+\cdot HO_4S^-$
M_r = 385.43
Monoclinic, $P2_1/c$
a = 12.2053 (5) Å
b = 9.4116 (4) Å
c = 20.2984 (7) Å
β = 126.7654 (15)°
V = 1867.91 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 293 (2) K
0.39 × 0.22 × 0.12 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003) T_{min} = 0.910, T_{max} = 0.976
61192 measured reflections
4441 independent reflections
3434 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.103
S = 1.02
4441 reflections
247 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82 (2)	1.86 (2)	2.6774 (16)	170 (2)
N1—H1*A*···O3^{ii}	0.86	2.00	2.8314 (16)	164
N2—H2···O2^{ii}	0.86	2.48	2.9123 (15)	112
N3—H3*A*···O4	0.86	1.92	2.7633 (15)	168

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

The anion H atom was located in a difference Fourier map and its coordinates were refined with U_{iso}(H) = 1.5U_{eq}(O). The other H atoms were placed at calculated positions and refined as riding on their parent atoms [C—H = 0.93 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,N)].

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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