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The First Structural Characterization of a Sulfoximidium Salt

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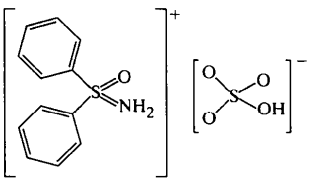
Abstract

The title compound, diphenylsulfoximidium hydrogen sulfate, $C_{12}H_{12}NOS^{+}.HSO_4^{-}$, was formed as a hydrolysis product during recrystallization of the product of the reaction of Ph_2SO with $(NSCl)_3$. Hydrogen-bonded networks link the cation and anion, the latter forming a hydrogen-bonded dimer.

Comment

It has been previously reported (Becke-Goehring & Latscha, 1962) that reaction of Me_2SO with $(NSCl)_3$ yielded the compound $[Me_2SNSMe_2][Cl]$. We have been interested in exploring this synthetic methodology to prepare new derivatives, including $[Ph_2SNSPh_2][Cl]$, which has been prepared previously by alternative routes (Furukawa *et al.*, 1973). In the course of our work investigating the reactivity of Ph_2SO with $(NSCl)_3$, we crystallized the title compound, (I), as a hydrolysis product. Hydrolysis probably occurs in the recrystallization for which the acetone solvent was not dried.



(I)

In (I), the cation is composed of a pseudo-tetrahedral S atom bonded to two phenyl groups, an amine group and an O atom (Fig. 1). The hydrogen sulfate counterion exhibits some disorder which has been modelled over three sites with site occupancies in the approximate ratio 0.74:0.17:0.09. Cations and anions are linked together through an N—H···O hydrogen-bonding network [N···O 2.824 (5) Å]. Hydrogen bonding also leads to the hydrogen sulfate anions forming dimers, with an O···O distance of 2.596 (8) Å. In addition, there is a close contact between an *ortho*-phenyl-H atom and a neighbouring O atom of a hydrogen sulfate anion [O5···C12 3.318 (5) Å].

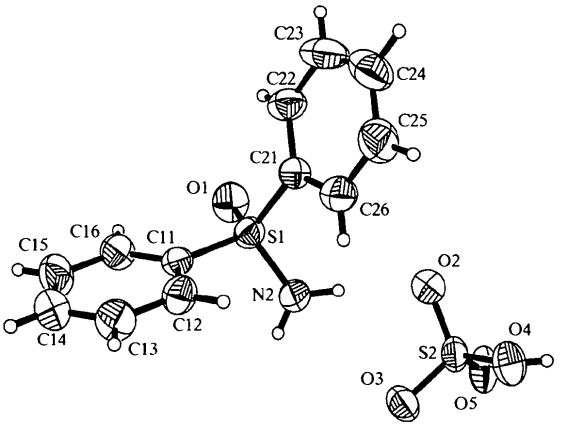


Fig. 1. The asymmetric unit of (I) showing the atom-labelling scheme and 50% probability displacement ellipsoids. Only the major component of the hydrogen sulfate disorder is shown for clarity.

Experimental

The synthesis of (I) was carried out by reaction of Ph_2SO and $(NSCl)_3$ in a 6:1 molar ratio in CCl_4. The solution was refluxed for 18 h and then cooled to room temperature. CCl_4 was removed *in vacuo* and the residue dissolved in acetone. Crystals suitable for X-ray diffraction formed over the course of 3–4 d.

Crystal data

$C_{12}H_{12}NOS^{+}.HSO_4^{-}$ — Mo $K\alpha$ radiation
M_r = 315.35 — λ = 0.71069 Å

Triclinic
$P\bar{1}$
a = 8.900 (2) Å
b = 11.508 (2) Å
c = 7.7675 (10) Å
α = 94.713 (14)°
β = 107.873 (13)°
γ = 67.784 (14)°
V = 700.4 (2) Å^3
Z = 2
D_x = 1.495 Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
θ = 30–40°
μ = 0.398 mm^{-1}
T = 293 (2) K
Block
0.30 × 0.25 × 0.20 mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6290 measured reflections
3203 independent reflections
2489 reflections with $I > 2\sigma(I)$

R_{int} = 0.018
θ_{max} = 27.51°
$h = -10 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 9$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.099
S = 1.026
3200 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.1658P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.267 e Å^{-3}
$\Delta\rho_{min}$ = −0.292 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—O1	1.436 (2)	S1—C11	1.756 (2)
S1—N2	1.564 (2)	S1—C21	1.761 (2)
O1—S1—N2	120.21 (11)	O1—S1—C21	110.17 (10)
O1—S1—C11	109.07 (9)	N2—S1—C21	102.92 (10)
N2—S1—C11	103.61 (10)	C11—S1—C21	110.44 (9)

The hydrogen sulfate anion was found to be disordered. Three sets of O atoms were refined with equivalent displacement parameters and idealized tetrahedral geometries. Aromatic H atoms were constrained with a riding model [$U_H = 1.2U_{iso}$(C)]. Both N—H and hydrogen sulfate H atoms (major component only) were located in a difference map and their coordinates refined with a fixed displacement parameter [$U_H = 1.2U_{iso}$(N) and $U_H = 0.08 \times 10^3$ Å^2, respectively].

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1214). Services for accessing these data are described at the back of the journal.

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Rotundifoline, an Oxoindole Alkaloid

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Abstract

In the title compound, methyl 2-{6′-ethyl-2′,3′,5′,6′,-7′,8′-hexahydro-4-hydroxy-2-oxo-spiro[1*H*-indole-3(2*H*),1′(8a′*H*)-indolizin]-7′-yl}-3-methoxyacrylate, $C_{22}H_{28}N_2O_5$, the indole molecule is not planar. The planarity of the atom group C13—N1—C2═O1 of the indole moiety and the short N1—C2 bond of 1.363 (11) Å are due to delocalization of the benzoid electrons, which extend over the atoms N1, C2 and O1. The five-membered ring of the indolizine moiety is puckered and the six-membered ring fused to it has a normal chair conformation. The methoxycarbonyl and the methoxy groups have a *trans* configuration about the C16═C17 bond in the acrylate moiety. The structure is stabilized by intramolecular hydrogen bonding of the type O—H···N and intermolecular hydrogen bonding of the type N—H···O.