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Redetermination of 4-nitrobenzene-sulfonamide

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Key indicators

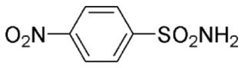
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.112
Data-to-parameter ratio = 16.1

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

In the crystal structure of the title compound, $C_6H_6N_2O_4S$, the nitro group is nearly coplanar with the aromatic ring, the dihedral angle being 1.6 (2)°. The overall molecular organization is stabilized by well defined intermolecular hydrogen bonds, leading to the formation of an infinite planar network structure.

Comment

It is known that the diuretically active title compound, (I), is an inhibitor of zinc–enzyme carbonic anhydrase which catalyses the reversible hydration of carbon dioxide (De Benedetti *et al.*, 1988). Its IR and NMR spectra have been reported previously (De Benedetti *et al.*, 1988; Pomerantz *et al.*, 1987; Dahn *et al.*, 1991). The crystal structure of (I) was originally analysed by Tremayne *et al.* (2002) from X-ray powder diffraction data using the Rietveld method. In addition, the crystal and molecular structure of the inclusion compound of the *cis–anti–cis* diastereomer of dicyclohexano-18-crown-6 with the title compound has been determined by X-ray analysis (Fonar *et al.*, 1992). In order to obtain more precise knowledge about the benzenesulfonamide compound, a single-crystal X-ray analysis of (I) was performed in our laboratory.



(I)

Fig. 1 shows a perspective view of the molecule, together with hydrogen-bonding connections to atoms in adjacent molecules. Angles C1—S1—N1 [107.99 (9)°], C1—S1—O1

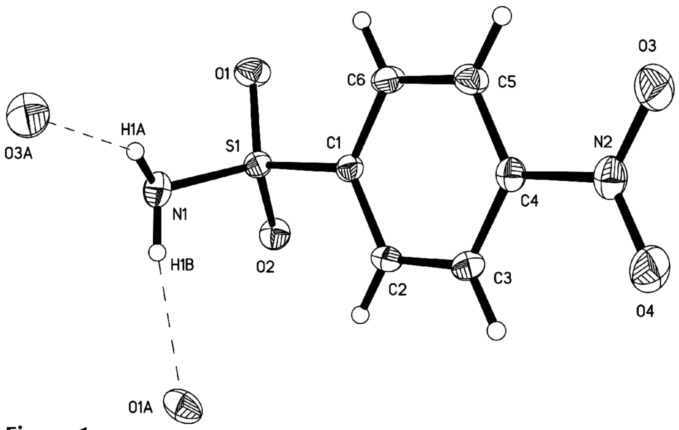


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. Dashed lines indicate hydrogen bonds. O1A and O3A correspond to O1^{ii} and O3^{i} in Table 1.

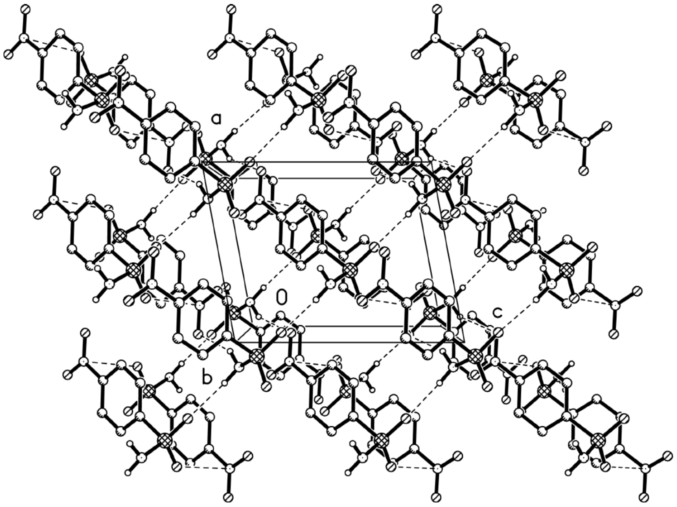


Figure 2
Packing diagram for the title compound. Hydrogen bonds are shown as dashed lines.

[106.84 (9)°] and C1—S1—O2 [107.88 (9)°] are much narrower than O1—S1—O2 [119.75 (10)°]. This behaviour may be due to the mutual repulsion between the lone-pair electrons of two O atoms in the sulfonamide group. On the other hand, the nitro group is nearly coplanar with the aromatic ring, the dihedral angle being 1.6 (2)°. One of the sulfonamide H atoms approaches the nitro O atom of an adjacent molecule at a distance of 2.20 Å, while the other sulfonamide H atom approaches the sulfonamide O atom of another molecule at a distance of 2.18 Å, indicating the existence of two hydrogen bonds (Table 1), leading to the formation of an infinite planar network structure (Fig. 2).

Experimental

The title compound was obtained from a commercial source (Aldrich). The crystal used for the data collection was obtained by slow evaporation of an ethanol solution at room temperature.

Crystal data

$C_6H_6N_2O_4S$
$M_r = 202.19$
Monoclinic, $P2_1/n$
$a = 7.068$ (1) Å
$b = 13.492$ (2) Å
$c = 8.841$ (1) Å
$\beta = 100.459$ (3)°
$V = 829.1$ (2) Å^3
$Z = 4$

$D_x = 1.620$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 3018 reflections
$\theta = 1$–27.5°
$\mu = 0.37$ mm^{-1}
$T = 294$ (2) K
Block, colourless
0.50 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.835$, $T_{\max} = 0.936$
5526 measured reflections

1901 independent reflections
1457 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 27.6°$
$h = -9 \rightarrow 8$
$k = -17 \rightarrow 17$
$l = -11 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.112$
$S = 1.05$
1901 reflections
118 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.1102P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.46$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3$^{\rm i}$	0.90	2.20	3.025 (2)	153
N1—H1B···O1$^{\rm ii}$	0.90	2.18	3.075 (3)	171

Symmetry codes: (i) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$.

H atoms were placed in geometrically calculated positions, with C—H = 0.93 and N—H = 0.90 Å, and were included in the final refinement in the riding-model approximation, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(C or N).

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *SHELXTL* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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