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2-(Toluene-4-sulfonylamino)benzoyl fluoride

Burkhard Ziemer,* Oxana Surygina and Max Ehwald

Institut für Chemie, Humboldt-Universität zu Berlin, Hessische Straße 1-2, 10115 Berlin, Germany

Correspondence e-mail: burkhard=ziemer@chemie.hu-berlin.de

Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.082
Data-to-parameter ratio = 13.1

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

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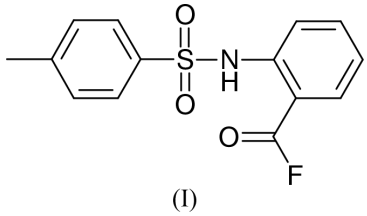
The title compound, $C_{14}H_{12}FNO_3S$, was used as an activating agent for a chemical synthesis, from which it recrystallized immediately. In the structure, one intramolecular hydrogen bond was found, which leads to the formation of a six-membered ring within one half of the molecule. In the other half of the molecule, there is a disubstituted benzene ring. The electron-withdrawing effect of the S atom and the electron-releasing effect of the opposite methyl substituent are both observed in this structure.

Comment

The title compound, (I), was used as an activating agent for N-containing heterocycles in order to produce iminium salts. During the reaction of those salts with nucleophiles, crystals of (I) suitable for X-ray studies were formed. In the structure of (I), one hydrogen bond between N—H and the acidic O atom was found (Fig. 1), leading to the formation of a six-membered ring. The sum of its endocyclic angles is 719.4 (2)°, *i.e.* near the ideal value of a planar hexagon. As a consequence, this portion of the molecule forms a common plane including the attached benzene ring and fluorine. The root-mean-square deviation of the atoms from this plane is 0.031 Å. The *para*-substituted benzene ring shows approximately the expected C_{2v} symmetry so that for the two matching endocyclic bond angles this holds true even at the 2σ level (Table 1). Therefore, it may be concluded that the angles at C1 [120.99 (13)°] and C4 [118.47 (14)°] are significantly enlarged and diminished, respectively. According to the conclusions of Domenicano *et al.* (1975), it follows that the S atom introduces a small electron-withdrawing effect in (I), whereas the methyl substituent introduces electron-releasing properties.



(I)

Experimental

2-(Toluene-4-sulfonylamino)benzoic acid, synthesized according to Nandi & Debnath (1978), reacts with 1.2 equivalents of cyanuric fluoride in dichloromethane at 263 K. After 1 h of reaction, the product was poured into ice water. From this mixture, the aqueous layer was extracted with dichloromethane. The combined organic phases were dried over $MgSO_4$ and evaporated under reduced pressure affording (I) in 93% yield.

Crystal data

$C_{14}H_{12}FNO_3S$
M_r = 293.31
Monoclinic, $P2_1/a$
a = 11.908 (3) Å
b = 8.2396 (14) Å
c = 13.925 (4) Å
β = 103.14 (3)°
V = 1330.6 (5) Å^3
Z = 4
D_x = 1.464 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5000 reflections
θ = 2.9–24.8°
μ = 0.26 mm^{-1}
T = 180 (2) K
Prism, colorless
0.60 × 0.36 × 0.28 mm

Data collection

Stoe IPDS diffractometer
φ-oscill., φ-incr. = 1.5°, 153 exposure scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
T_{min} = 0.859, T_{max} = 0.931
8860 measured reflections
2439 independent reflections
2048 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.8°
$h = -14 \rightarrow 14$
$k = -9 \rightarrow 9$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.082
S = 1.04
2439 reflections
186 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.2945P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.009
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (°).

C6—C1—C2	120.99 (13)	C5—C4—C3	118.47 (14)
C3—C2—C1	118.91 (14)	C4—C5—C6	121.05 (14)
C2—C3—C4	121.42 (15)	C1—C6—C5	119.14 (14)

Methyl was refined as a rigid group, the other C-bound H atoms as riding. The coordinates of the N-bound H atom were freely refined, but its displacement parameter was constrained to equal that of N.

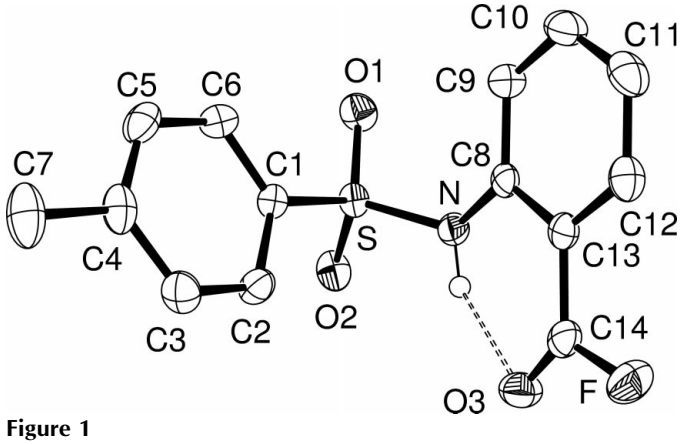


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids (Farrugia, 1997). Only the N-bound H atom is shown.

Data collection: *IPDS*2.87 (Stoe & Cie, 1997); cell refinement: *IPDS*2.87; data reduction: *IPDS*2.87; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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