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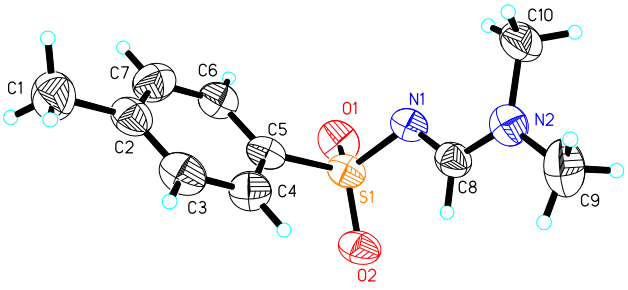
Crystal structure of *N*, *N*-Dimethyl-*N'*-tosylformimidamide, $C_{10}H_{14}N_2O_2S$



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Abstract

$C_{10}H_{14}N_2O_2S$, Triclinic, $P\bar{1}$ (no. 2), a = 7.622(3) Å, b = 7.949(2) Å, c = 10.868(3) Å, α = 74.179(4)°, β = 78.292(4)°, γ = 62.651(4)°, V = 560.4(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0498, w$R_{ref}(F^2)$ = 0.1424, T = 293(2) K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

1 Source of materials

According to the synthesis method in refs. 4–6, NBS (196 mg, 1.1 mmol) and water (3.6 μL) were added to a solution of 4-methylbenzenesulfonamide (171 mg, 1.0 mmol) in DMF (2 mL). The resulting solution was stirred at 80 °C for 8 h. After completion of the reaction, the mixture was extracted with dichloromethane (3 × 10 mL) and the combined extracts were dried with anhydrous sodium sulfate. The solvent was removed under reduced pressure, then the pure product was purified by silica gel column chromatography to obtain the pure white solid. The colorless crystals of *N*, *N*-Dimethyl-*N'*-tosylformimidamide were obtained by slow evaporation of a dichloromethane solution at room temperature.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.05 × 0.05 × 0.04 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.27 mm^{-1}
Diffractometer, scan mode:	Bruker Apex2, φ and ω scans
θ_{max}, completeness:	25.1°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3988, 1966, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1,446
$N(param)_{refined}$:	139
Programs:	Bruker,[1] Olex2,[2] Shelx[3]

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and refined isotropically using a riding model. For all C(H) groups, U_{iso}(H) = 1.2 U_{eq}(C); for all C(H, H, H) groups, U_{iso}(H) = 1.5 U_{eq}(C). Idealised Me was refined as rotating group.

3 Comment

The synthesis of N-sulfonylamidines has attracted a great deal of attention due to their widespread applications in organic synthesis and biochemistry.[7–11] Due to their unique structural motifs, they exhibit a range of biological activities like antibacterial, antifungal, and anticancer properties,[12,13] and also play an important role in delaying Alzheimer's disease.[14]

The title structure crystallized in the triclinic $P\bar{1}$ space group, with one molecule in the asymmetric unit. In the crystal structure, a methyl formate group and a sulfamidine group are connected to the same benzene ring with C(1)–C(2) and C(5)–S(1). The bond lengths of S(1)–C(5), S(1)–O(1), S(1)–O(2), and S(1)–N(1) are 1.750(3), 1.429(2), 1.437(2), 1.611(2) Å, respectively. The bond lengths of N(1)–C(8), N(2)–C(8), N(2)–C(10) and N(2)–C(9) are 1.297(4), 1.310(4), 1.450(4) and 1.447(4) Å, respectively. The bond angles of C(5)–S(1)–N(1), C(5)–S(1)–O(2),

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C(5)–S(1)–O(1), N(1)–S(1)–O(1), N(1)–S(1)–O(2) and O(1)–S(1)–O(2) are 103.42(13), 107.94(13), 108.57(14), 107.26(13), 112.12(13) and 116.69(13)°. The bond angles of N(1)–C(8)–N(2), C(8)–N(2)–C(9) and C(9)–N(2)–C(10) are 122.9(3), 122.2(3) and 117.1(3)°. All geometrical parameters are comparable with those of structural analogs that were found in the Cambridge Structural Database.[15]

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