Crystal structure of *N*,*N*-dimethyl-2-sulfamoylnicotinamide, $C_8H_{11}N_3O_3S$

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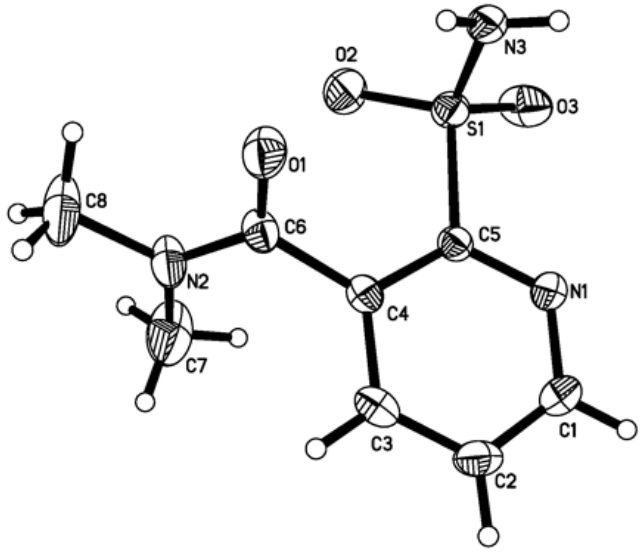

Abstract

$C_8H_{11}N_3O_3S$, triclinic, $P\bar{1}$ (no. 2), a = 7.819(1) Å, b = 8.042(1) Å, c = 9.551(1) Å, α = 91.260(1)°, β = 90.918(1)°, γ = 112.659(1)°, V = 553.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.104, T = 273 K.

Source of material

To a solution of 3-(dimethylcarbamoyl)pyridine-2-sulfonyl chloride (50 mmol) in anhydrous dichloromethane (50 ml) was added ammonia (120 mmol). After stirring the reaction mixture for 6 h at room temperature, 100 ml water was added. The oil fraction after separation was concentrated under reduced pressure and the residue was recrystallized from dichloromethane to give the title compound in a yield of 63 % [1]. Crystals suitable for X-ray diffraction were obtained by recrystallization from dichloromethane/pentane (1:1) at room temperature in a yield of 72 %.

Experimental details

All H atoms on C atoms were placed in idealized positions with d(C—H) = 0.96 (methyl), 0.97 (methylene) and 0.93 Å (aromatic) and included in the refinement in the riding-model approximation with U_{iso}(H) = 1.5 U_{eq}(C_{methyl}) and 1.2 U_{eq}($C_{methylene, aromatic}$). H atoms of sulfonamide were located in a difference Fourier map and refined freely. This was done because of uncertainty about the hybridization of the N atom, so that position constraints would possibly have led to a wrong model.

Discussion

The title compound, used for preparation of Nicosulfuron, which is a member of the sulfonylurea family of herbicides and recommended for controlling Johnsongrass (Sorghum halepense) in maize [2].

In the title crystal structure, the dihedral angle between the amide group and pyridine ring is 65.06(9)°. The bond distances and bond angles are all in normal range. The crystal structure features intermolecular N–H···O and C–H···O hydrogen bonds, leading to the formation of a 3D framework.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.18 × 0.20 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.84 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3716, 1959
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1692
$N(param)_{refined}$:	146
Programs:	SHELXS-97, SHELXL-97, SHELXTL [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.4577	–0.1668	0.7823	0.054
H(1)	2*i*	0.8519	0.3055	0.8939	0.061
H(2)	2*i*	0.7644	0.0042	0.8437	0.060
H(7A)	2*i*	0.3184	–0.0343	0.5380	0.138
H(7B)	2*i*	0.1397	–0.1498	0.4495	0.138
H(7C)	2*i*	0.2581	–0.2438	0.5226	0.138
H(8A)	2*i*	–0.0998	–0.4111	0.6808	0.132
H(8B)	2*i*	–0.1295	–0.3145	0.5472	0.132
H(8C)	2*i*	–0.1653	–0.2504	0.6961	0.132
H(3NA)	2*i*	0.139(4)	0.248(4)	0.982(3)	0.066(9)
H(3NB)	2*i*	0.280(4)	0.420(4)	1.019(3)	0.057(7)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*	0.29306(7)	0.35037(6)	0.80978(6)	0.0397(3)	0.0334(3)	0.0516(3)	0.0167(2)	–0.0008(2)	0.0023(2)
N(1)	2*i*	0.6126(2)	0.3226(2)	0.8571(2)	0.0339(9)	0.039(1)	0.053(1)	0.0113(7)	–0.0017(8)	–0.0052(8)
O(1)	2*i*	0.0651(2)	–0.0816(2)	0.8738(2)	0.0450(9)	0.062(1)	0.0470(9)	0.0052(7)	0.0099(7)	–0.0071(7)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(5)	2*i*	0.4380(3)	0.2239(2)	0.8201(2)	0.034(1)	0.031(1)	0.035(1)	0.0130(8)	0.0015(8)	0.0001(8)
C(4)	2*i*	0.3689(3)	0.0387(2)	0.7918(2)	0.040(1)	0.032(1)	0.032(1)	0.0128(8)	0.0032(8)	0.0004(8)
N(3)	2*i*	0.2048(3)	0.3470(3)	0.9582(2)	0.034(1)	0.040(1)	0.059(1)	0.0110(8)	0.0061(9)	–0.005(1)
C(6)	2*i*	0.1648(3)	–0.0749(3)	0.7741(2)	0.044(1)	0.032(1)	0.039(1)	0.0088(9)	0.0029(9)	–0.0021(8)
N(2)	2*i*	0.1042(3)	–0.1706(2)	0.6552(2)	0.051(1)	0.047(1)	0.045(1)	–0.0009(9)	0.0056(8)	–0.0106(8)
O(2)	2*i*	0.1487(2)	0.2570(2)	0.7100(2)	0.063(1)	0.071(1)	0.064(1)	0.0374(9)	–0.0250(9)	–0.0100(9)
C(3)	2*i*	0.4960(3)	–0.0436(3)	0.8011(2)	0.058(1)	0.036(1)	0.045(1)	0.024(1)	0.007(1)	–0.0003(9)
O(3)	2*i*	0.4126(2)	0.5323(2)	0.7865(2)	0.065(1)	0.0354(8)	0.096(1)	0.0219(8)	0.024(1)	0.0191(8)
C(1)	2*i*	0.7294(3)	0.2382(3)	0.8666(2)	0.035(1)	0.058(1)	0.060(1)	0.019(1)	–0.003(1)	–0.004(1)
C(2)	2*i*	0.6780(3)	0.0574(3)	0.8381(2)	0.051(1)	0.059(1)	0.052(1)	0.036(1)	0.002(1)	0.002(1)
C(7)	2*i*	0.2145(4)	–0.1477(5)	0.5308(3)	0.073(2)	0.121(3)	0.051(2)	0.004(2)	0.011(1)	–0.029(2)
C(8)	2*i*	–0.0893(4)	–0.2976(4)	0.6439(3)	0.066(2)	0.080(2)	0.073(2)	–0.019(2)	0.000(2)	–0.022(2)

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