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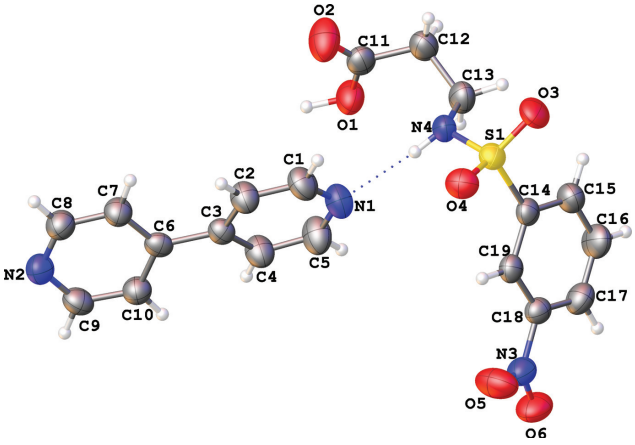
Crystal structure of 3-((3-nitrophenyl) sulfonamido)propanoic acid — 4,4′-bipyridine (1/1), $C_{19}H_{18}N_4O_6S$



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Abstract

$C_{19}H_{18}N_4O_6S$, monoclinic, $P2_1/n$ (no. 14), $a = 8.343(4)$ Å, $b = 7.367(3)$ Å, $c = 31.532(14)$ Å, $\beta = 97.398(8)°$, $V = 1921.8(14)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0420$, $wR_{ref}(F^2) = 0.1158$, $T = 296.15$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.32 × 0.21 × 0.17 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.22 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9298, 3372, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2569
$N(param)_{refined}$:	279
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
S1	0.77366(6)	0.04750(8)	0.66439(2)	0.04207(19)
O1	1.1862(2)	0.4214(3)	0.60436(5)	0.0626(5)
O2	0.9970(2)	0.4060(3)	0.54952(6)	0.0787(6)
O3	0.63382(18)	0.1455(2)	0.67080(5)	0.0594(5)
O4	0.75810(18)	−0.1214(2)	0.64291(5)	0.0525(4)
O5	1.0997(3)	−0.4700(3)	0.74676(7)	0.0811(6)
O6	1.2458(2)	−0.3503(3)	0.79975(6)	0.0699(5)
N1	0.8796(2)	0.0477(3)	0.39833(6)	0.0563(5)
N2	0.3723(2)	0.3264(3)	0.54736(6)	0.0507(5)
N3	1.1397(2)	−0.3428(3)	0.76985(6)	0.0535(5)
N4	0.8818(2)	0.1731(3)	0.63859(5)	0.0434(5)
C1	0.9365(3)	0.1019(4)	0.43682(8)	0.0568(7)
H1A	1.047867	0.098111	0.444539	0.068*
C2	0.8439(3)	0.1636(3)	0.46652(7)	0.0505(6)
H2	0.891998	0.200824	0.493350	0.061*
C3	0.6797(2)	0.1698(3)	0.45631(6)	0.0389(5)
C4	0.6202(3)	0.1195(3)	0.41567(7)	0.0529(6)
H4A	0.509697	0.125984	0.406676	0.063*
C5	0.7221(3)	0.0600(4)	0.38829(8)	0.0625(7)
H5	0.677773	0.025984	0.360869	0.075*
C6	0.5732(2)	0.2249(3)	0.48772(7)	0.0392(5)
C7	0.6236(3)	0.2233(3)	0.53093(7)	0.0478(6)
H7	0.728597	0.187618	0.540971	0.057*
C8	0.5221(3)	0.2731(3)	0.55904(8)	0.0525(6)
H8	0.560129	0.269434	0.588075	0.063*
C9	0.3216(3)	0.3299(3)	0.50611(8)	0.0502(6)
H9	0.216515	0.368457	0.497190	0.060*
C10	0.4160(3)	0.2795(3)	0.47563(7)	0.0472(6)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H10	0.374036	0.282188	0.446849	0.057*
C11	1.0373(3)	0.4323(3)	0.58653(8)	0.0489(6)
C12	0.9215(3)	0.4835(3)	0.61636(8)	0.0544(6)
H12A	0.813658	0.487489	0.600651	0.065*
H12B	0.947653	0.604583	0.627177	0.065*
C13	0.9209(3)	0.3576(3)	0.65322(7)	0.0534(6)
H13A	1.026371	0.358728	0.670247	0.064*
H13B	0.841980	0.398511	0.671170	0.064*
C14	0.8822(2)	0.0093(3)	0.71510(6)	0.0387(5)
C15	0.8844(3)	0.1381(3)	0.74662(7)	0.0536(6)
H15	0.824476	0.244177	0.741619	0.064*
C16	0.9748(3)	0.1101(4)	0.78531(8)	0.0622(7)
H16	0.977005	0.198283	0.806472	0.075*
C17	1.0619(3)	−0.0459(4)	0.79335(7)	0.0538(6)
H17	1.124269	−0.065302	0.819601	0.065*
C18	1.0544(2)	−0.1722(3)	0.76157(7)	0.0414(5)
C19	0.9670(2)	−0.1482(3)	0.72244(6)	0.0397(5)
H19	0.965133	−0.236698	0.701349	0.048*
H1	1.259(4)	0.382(4)	0.5824(11)	0.107(11)*
H4	0.957(3)	0.109(3)	0.6299(7)	0.050(7)*

Source of material

To a 18 mL mixed solution of *N*,*N*-dimethylformamide and water (1:1.5) was added $Mn(CH_3COO)_2 \cdot 4H_2O$ (122.5 mg, 0.5 mmol), 3-((3-nitrophenyl)sulfonamido)propanoic acid (274.3 mg, 1.0 mmol) and 4,4′-bipyridine (156.2 mg, 1.0 mmol). Then the pH of the mixture was adjusted to 7 and added to a 20 mL Teflon-lined reaction vessel. After heating at 80 degrees for 24 h, it was filtrated and naturally evaporated for one week to give slightly yellow block crystals as an unexpected product.

Experimental details

Hydrogen atoms were positioned geometrically. Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms. The structure was solved with the ShelXT [3] structure solution program using Intrinsic Phasing and refined with the ShelXL [4] refinement package.

Comment

Sulfonamide and its derivatives have attracted wide attention due to their various biological activities, such as antibacterial [5, 6], antiviral [7, 8], antimalarial [9, 10] and anticancer [8, 11]. In addition, N and O atoms in the structure can be used as electron donors for coordination bonds, which may result in different coordination modes [12, 13], which facilitates the construction of metal-organic coordination polymers. In recent years, sulfonamide and its derivatives have been widely used to construct metal-organic coordination polymers [14, 15]. A new crystal structure of a sulfonamide compound was unexpectedly obtained when we were trying to construct metal-organic coordination polymers.

The asymmetric unit of the title complex contains two independent molecules, 3-((3-nitrophenyl)sulfonamido) propanoic acid and 4,4′-bipyridine, respectively. The extended 3D network is formed through $\pi\cdots\pi$ stacking interactions between 4,4′-bipyridine molecules (centroid-centroid separations: 3.535 Å) and hydrogen bonds ($O1\cdots N2^{\#1}$: 2.617(3) Å, O1-H1$\cdots$N2$^{\#1}$ angle 178.3°; $N4\cdots N1^{\#2}$: 2.926(3) Å, N4-H4$\cdots$N1$^{\#2}$ angle 173.2°; symmetry code: #1 = 1 + *x*, +*y*, +*z*; #2 = 2 − *x*, −*y*, 1 − *z*) through carboxylic oxygen atoms, amino nitrogen atoms and pyridine nitrogen atoms. All bond lengths and angles are within the expectations.

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