Crystal structure of *cis*-2,5-bis(3-pyridyl)-1,3,4-thiadiazole, $C_{12}H_8N_4S$

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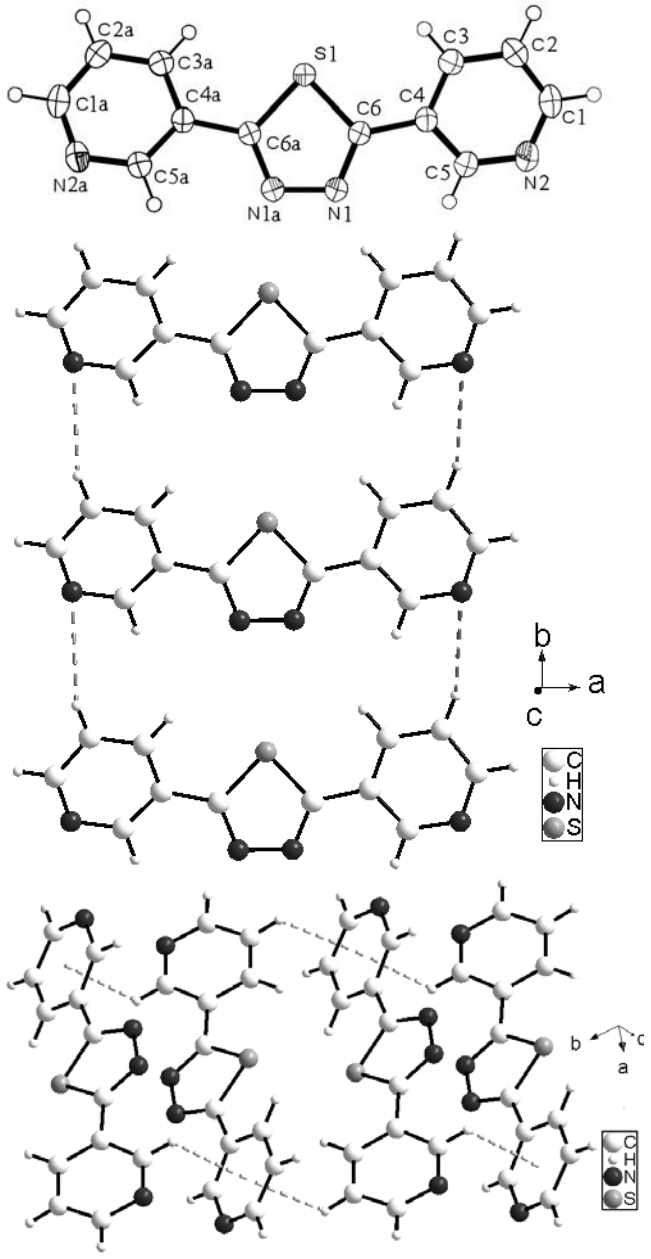

Abstract

$C_{12}H_8N_4S$, monoclinic, *C*12/*c*1 (no. 15), a = 26.367(4) Å, b = 5.7703(8) Å, c = 7.136(1) Å, β = 102.884(2)°, V = 1058.4 Å^3, Z = 1, $R_{gt}(F)$ = 0.032, $wR_{ref}(F^2)$ = 0.096, T = 173 K.

Source of material

2,5-Bis(3-pyridyl)-1,3,4-thiadiazole was prepared similarly to the literature procedure [1]. A mixture of nickel acetate monohydrate (0.026 g, 0.1 mmol), 2,5-bis(3-pyridyl)-1,3,4-thiadiazole (0.024 g, 0.1mmol), 2,2-dipy-4,4-bicarboxylic acid (0.012 g, 0.05 mmol), ethanol (5 ml) and 25 % ammonia water (3 ml) was sealed in a 15 ml Teflon-lined stainless steel reactor. The reactor was heated in an oven at 180 °C for 72 hours and then cooled to room temperature at a rate of 10 °C/h. Yellow needle-like crystals were obtained.

Discussion

The crystal structure of *trans*-2,5-bis(3-pyridyl)-1,3,4-thiadiazole has been already reported [2], and the structures of its other isomers (4-pyridyl or 2-pyridyl-1,3,4-thiadiazole) have been previously X-ray characterized [3-5].

The two pyridyl rings within each molecule (figure, top) form a dihedral angle of 20.78(5)° with the mean plane difined by the central thiadiazole ring, which is larger than those in the *trans*-2,5-bis(3-pyridyl)-1,3,4-thiadiazole molecule. There is a 2-fold axis through the middle point of the N—N bond in the thiadiazole ring and the sulfur atom. There are C–H···N hydrogen bonds between *cis*-2,5-bis(3-pyridyl)-1,3,4-thiadiazole molecules with the distance of H···A about 2.95 Å (figure, middle). Another kind of intermolecular interaction is C–H···π interactions (figure, bottom). The distance between hydrogen atom and the center of aromatic pyridyl ring is about 2.8 Å. Furthermore, it is interesting that *cis*- and *trans*-2,5-bis(3-pyridyl)-1,3,4-thiadiazole are synthesized under very different conditions. This indicates that factors like temperature, metal ions, other organic compounds in the reaction mixture may affect the conformation of the organic molecules.

Table 1. Data collection and handling.

Crystal:	yellow needle, size 0.09 × 0.11 × 0.54 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.85 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II CCD, φ/ω
$2\theta_{max}$:	54.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3265, 1212
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1058
$N(param)_{refined}$:	78
Programs:	SHELXS-97 [6], SHELXL-97 [7], SHELXTL [8]

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

Atom	Site	x	y	z	U_{iso}
H(1)	8*f*	0.2343	0.2095	0.7093	0.055
H(2)	8*f*	0.1753	–0.0885	0.6193	0.050
H(3)	8*f*	0.0878	–0.0296	0.6289	0.044
H(4)	8*f*	0.1267	0.6136	0.8306	0.046

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}
C(1)	8*f*	0.19909(6)	0.2338(3)	0.7162(2)	0.0258(7)	0.052(1)	0.063(1)	0.0032(7)	0.0153(7)	0.0000(8)
C(2)	8*f*	0.16418(6)	0.0554(3)	0.6609(2)	0.0356(7)	0.0380(8)	0.0532(9)	0.0080(6)	0.0143(7)	–0.0016(7)
C(3)	8*f*	0.11274(5)	0.0898(3)	0.6669(2)	0.0298(7)	0.0324(7)	0.0484(8)	–0.0009(6)	0.0076(6)	–0.0039(6)
C(4)	8*f*	0.09823(5)	0.3022(2)	0.7295(2)	0.0252(6)	0.0306(7)	0.0383(7)	0.0008(5)	0.0086(5)	0.0028(5)
C(5)	8*f*	0.13648(5)	0.4693(3)	0.7853(2)	0.0301(7)	0.0329(8)	0.0553(9)	–0.0025(6)	0.0142(6)	–0.0038(6)
C(6)	8*f*	0.04417(5)	0.3526(2)	0.7398(2)	0.0245(6)	0.0271(7)	0.0386(7)	–0.0015(5)	0.0078(5)	0.0000(5)
N(1)	8*f*	0.02565(4)	0.5619(2)	0.7439(2)	0.0267(6)	0.0286(6)	0.0614(8)	–0.0014(5)	0.0144(5)	0.0007(5)
N(2)	8*f*	0.18625(5)	0.4385(3)	0.7788(2)	0.0289(6)	0.0464(8)	0.073(1)	–0.0052(6)	0.0171(6)	–0.0060(7)
S(1)	4*e*	0	0.13583(8)	¾	0.0258(3)	0.0252(3)	0.0582(4)	0	0.0133(2)	0

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