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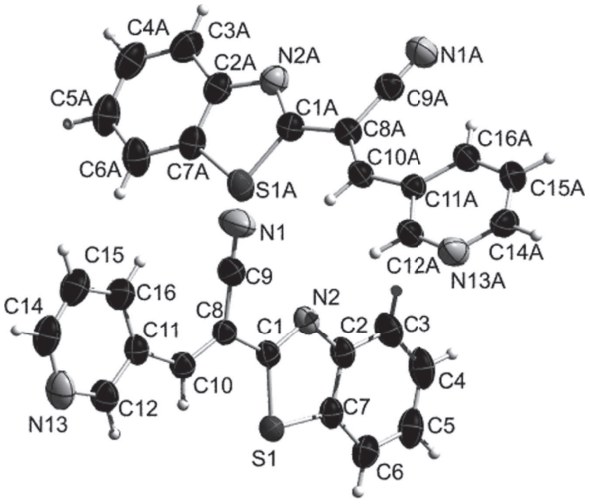
Crystal structure of (*E*)-2-(benzo[*d*]thiazol-2-yl)-3-(pyridin-3-yl)acrylonitrile)



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Abstract

$C_{15}H_9N_3S$, triclinic, $P\bar{1}$ (no. 2), $a = 9.5737(5)$ Å, $b = 12.0958(4)$ Å, $c = 12.2705(7)$ Å, $\alpha = 64.083(5)$ Å, $\beta = 80.907(4)$ Å, $\gamma = 82.800(4)$, $V = 1259.44(11)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0460$, $wR_{ref}(F^2) = 0.140$, $T = 293(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Yellow, Block, size 0.16×0.18×0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.42 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction CCD area detector diffractometer, ω scans
$2\theta_{max}$:	52.4°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8631, 4877
$N(param)_{refined}$:	343
Programs:	SHELXL [18], Crysalis^{pro} [19]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(13A)	2*i*	0.2272	−0.2670	0.5731	0.054
H(10)	2*i*	0.7647	0.0826	0.5041	0.046
H(12A)	2*i*	0.4466	−0.2589	0.4760	0.060
H(6)	2*i*	0.3931	0.3229	0.1448	0.059
H(16A)	2*i*	0.3590	−0.4720	0.3255	0.057
H(3)	2*i*	0.4526	−0.0756	0.1642	0.064
H(10A)	2*i*	0.6234	−0.2929	0.3503	0.050
H(3A)	2*i*	1.1363	−0.4874	0.0575	0.074
H(13)	2*i*	1.0011	0.0517	0.7973	0.059
H(16)	2*i*	0.9348	−0.2259	0.6459	0.064
H(12)	2*i*	0.8619	0.1053	0.6491	0.060
H(14A)	2*i*	0.0709	−0.3755	0.5466	0.064
H(15A)	2*i*	0.1308	−0.4779	0.4254	0.064
H(6A)	2*i*	1.1673	−0.1465	0.1354	0.069
H(15)	2*i*	1.0793	−0.2793	0.7984	0.072
H(14)	2*i*	1.1048	−0.1389	0.8720	0.075
H(4)	2*i*	0.3102	0.0762	0.0304	0.071
H(5)	2*i*	0.2770	0.2702	0.0242	0.067
H(5A)	2*i*	1.3609	−0.2227	0.0449	0.074
H(4A)	2*i*	1.3454	−0.3900	0.0062	0.077

The crystal structure is shown in the figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*	0.58963(5)	0.16332(4)	0.33087(5)	0.0389(3)	0.0404(3)	0.0460(3)	−0.0018(2)	−0.0093(2)	−0.0226(2)
S(1A)	2*i*	0.87375(6)	−0.23778(6)	0.22050(6)	0.0411(3)	0.0644(4)	0.0577(3)	−0.0140(2)	0.0068(2)	−0.0376(3)
N(2)	2*i*	0.5936(2)	−0.0489(2)	0.3238(2)	0.0430(9)	0.0444(9)	0.0411(9)	−0.0074(7)	−0.0063(7)	−0.0222(7)
N(2A)	2*i*	0.8909(2)	−0.4237(2)	0.1643(2)	0.044(1)	0.050(1)	0.049(1)	−0.0023(8)	0.0004(8)	−0.0224(8)
C(13A)	2*i*	0.2533(2)	−0.3076(2)	0.5231(2)	0.039(1)	0.045(1)	0.061(1)	−0.0066(8)	0.0088(9)	−0.034(1)
C(1)	2*i*	0.6411(2)	0.0067(2)	0.3793(2)	0.0333(9)	0.042(1)	0.038(1)	−0.0071(7)	−0.0005(7)	−0.0195(8)
C(2)	2*i*	0.5085(2)	0.0328(2)	0.2377(2)	0.039(1)	0.051(1)	0.036(1)	−0.0111(8)	−0.0011(8)	−0.0202(9)
C(10)	2*i*	0.7880(2)	−0.0015(2)	0.5324(2)	0.037(1)	0.042(1)	0.040(1)	−0.0031(8)	−0.0039(8)	−0.0198(8)
C(12A)	2*i*	0.3825(2)	−0.3038(2)	0.4649(2)	0.048(1)	0.049(1)	0.062(1)	−0.0099(9)	0.000(1)	−0.032(1)
C(8)	2*i*	0.7316(2)	−0.0574(2)	0.4772(2)	0.0327(9)	0.041(1)	0.039(1)	−0.0042(7)	−0.0014(7)	−0.0196(8)
N(1A)	2*i*	0.5657(2)	−0.5378(2)	0.1888(2)	0.066(1)	0.068(1)	0.085(2)	−0.008(1)	−0.006(1)	−0.050(1)
C(9A)	2*i*	0.6061(2)	−0.4745(2)	0.2217(2)	0.045(1)	0.047(1)	0.051(1)	−0.0043(9)	−0.0005(9)	−0.026(1)
C(9)	2*i*	0.7527(2)	−0.1874(2)	0.5126(2)	0.050(1)	0.048(1)	0.054(1)	−0.0016(9)	−0.013(1)	−0.026(1)
C(7)	2*i*	0.4916(2)	0.1526(2)	0.2296(2)	0.0327(9)	0.050(1)	0.037(1)	−0.0089(8)	0.0009(7)	−0.0186(9)
C(1A)	2*i*	0.8051(2)	−0.3620(2)	0.2140(2)	0.042(1)	0.046(1)	0.038(1)	−0.0026(8)	−0.0044(8)	−0.0185(9)
C(11A)	2*i*	0.4301(2)	−0.3616(2)	0.3884(2)	0.039(1)	0.0353(9)	0.040(1)	−0.0038(7)	−0.0032(8)	−0.0159(8)
C(6)	2*i*	0.4047(2)	0.2435(2)	0.1493(2)	0.040(1)	0.056(1)	0.045(1)	−0.0053(9)	−0.0046(9)	−0.014(1)
C(7A)	2*i*	1.0348(2)	−0.2730(2)	0.1518(2)	0.039(1)	0.063(1)	0.038(1)	−0.0043(9)	−0.0023(8)	−0.023(1)
C(11)	2*i*	0.8798(2)	−0.0518(2)	0.6292(2)	0.0348(9)	0.049(1)	0.037(1)	−0.0072(8)	−0.0026(8)	−0.0195(9)
C(16A)	2*i*	0.3331(2)	−0.4300(2)	0.3743(2)	0.044(1)	0.049(1)	0.056(1)	−0.0085(9)	−0.0021(9)	−0.029(1)
C(3)	2*i*	0.4407(3)	0.0031(2)	0.1614(2)	0.057(1)	0.066(1)	0.046(1)	−0.015(1)	−0.008(1)	−0.028(1)
C(2A)	2*i*	1.0238(2)	−0.3747(2)	0.1291(2)	0.042(1)	0.055(1)	0.042(1)	−0.0010(9)	−0.0030(9)	−0.017(1)
C(10A)	2*i*	0.5769(2)	−0.3451(2)	0.3326(2)	0.041(1)	0.042(1)	0.045(1)	−0.0078(8)	−0.0036(8)	−0.0212(9)
C(3A)	2*i*	1.1418(3)	−0.4197(3)	0.0734(3)	0.049(1)	0.066(2)	0.065(2)	0.004(1)	0.002(1)	−0.030(1)
C(8A)	2*i*	0.6570(2)	−0.3916(2)	0.2600(2)	0.041(1)	0.040(1)	0.040(1)	−0.0044(8)	−0.0042(8)	−0.0172(8)
C(13)	2*i*	0.9874(2)	−0.0040(2)	0.7671(2)	0.047(1)	0.068(1)	0.044(1)	−0.006(1)	−0.0145(9)	−0.031(1)
C(16)	2*i*	0.9474(3)	−0.1697(2)	0.6758(2)	0.050(1)	0.057(1)	0.055(1)	0.001(1)	−0.013(1)	−0.025(1)
C(12)	2*i*	0.9055(2)	0.0263(2)	0.6794(2)	0.052(1)	0.058(1)	0.048(1)	−0.005(1)	−0.010(1)	−0.027(1)
N(1)	2*i*	0.7671(3)	−0.2912(2)	0.5434(3)	0.095(2)	0.048(1)	0.102(2)	0.005(1)	−0.041(2)	−0.036(1)
C(14A)	2*i*	0.1625(2)	−0.3720(2)	0.5069(2)	0.039(1)	0.046(1)	0.069(2)	−0.0008(9)	0.005(1)	−0.024(1)
C(15A)	2*i*	0.1978(2)	−0.4338(2)	0.4342(2)	0.043(1)	0.050(1)	0.071(2)	−0.0109(9)	−0.004(1)	−0.028(1)
C(6A)	2*i*	1.1608(3)	−0.2145(3)	0.1203(2)	0.046(1)	0.081(2)	0.049(1)	−0.017(1)	0.001(1)	−0.031(1)
C(15)	2*i*	1.0327(3)	−0.2016(3)	0.7665(2)	0.048(1)	0.069(2)	0.052(1)	0.002(1)	−0.013(1)	−0.016(1)
C(14)	2*i*	1.0482(3)	−0.1159(3)	0.8096(2)	0.046(1)	0.099(2)	0.042(1)	−0.009(1)	−0.011(1)	−0.026(1)
C(4)	2*i*	0.3557(3)	0.0942(3)	0.0818(2)	0.053(1)	0.086(2)	0.042(1)	−0.020(1)	−0.010(1)	−0.025(1)
C(5)	2*i*	0.3368(2)	0.2117(3)	0.0771(2)	0.042(1)	0.076(2)	0.040(1)	−0.010(1)	−0.0084(9)	−0.013(1)
C(5A)	2*i*	1.2754(3)	−0.2602(3)	0.0663(2)	0.040(1)	0.090(2)	0.050(1)	−0.012(1)	0.001(1)	−0.025(1)
C(4A)	2*i*	1.2662(3)	−0.3612(3)	0.0430(2)	0.041(1)	0.084(2)	0.059(2)	0.004(1)	0.004(1)	−0.028(1)

Source of material

The compound was obtained by Knoevenagel condensation between equimolar amounts of 2-(benzo[*d*]thiazol-2-yl)acetonitrile and 3-pyridinecarboxaldehyde according to literature [1]. The resulting mixtures were stirred for 18 minutes at room temperature using ethanol as solvent and catalytic amounts of triethylamine (TEA). The precipitates which formed were collected by filtration, washed with ethanol and dried, and then crystallized from ethanol giving the compound in yields of 66%; m.p. 155–157 °C; EI—MS (*m/z*): 263 (M+, 100), 226 (20), 210 (38). The ^{1}H-NMR analysis of this derivative revealed a single olefinic proton at δ 8.51 associated with the formation of a single *E*-isomer. Crystals suitable for single-crystal X-ray diffraction were grown from solutions in ethanol.

Experimental details

H atoms were located in the difference Fourier map, but refined with fixed individual displacement parameters, using a riding mode with C—H distances of 0.93 Å with U_{iso}(H) values of $1.2U_{eq}$(C).

Discussion

The heteroaryl-acrylonitriles have emerged recently as a new family of acetylcholinesterase inhibitors (AChEIs) [1, 2]. In addition, this family of compounds have been proved for having

prominent biological properties as antifungal, antitumor, and antibacterial activities [3–7], as well as applications for the design of dendrimers [8] and fluorescent probes for visualizing endogenous thiols in living cells [9, 10]. They have also been reported in the literature as versatile building-blocks for molecules with potential biological or pharmaceutical applications [11–17].

In the figure there are represented two molecules in the asymmetric unit, which are connected by non-classical C—H···N hydrogen bond interactions. The crystal structure is further stabilized by weak π-π stacking interactions with distance Cg1—Cg2^{i} 3.80 Å (Cg1: C11/C16; Cg2^{i}: C2/C7, symmetry code (i): $1-x$, $-y$, $1-z$). The main difference between both molecules is the dihedral angle formed by the benzothiazolyl fragment and the pyridine ring, which showed values of 4.97(6)° and 17.59(7)° for molecules A and B, respectively. The bond lengths and angles are in the expected ranges.

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