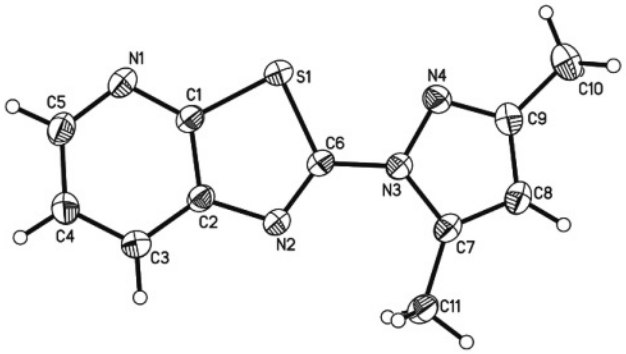
Crystal structure of 2-(3,5-dimethyl-1*H*-pyrazol-1-yl)thiazolo[5,4-*b*]-pyridine, $C_{11}H_{10}N_4S$

Yun-Lai Ren[*, I], Qian Wang[I], Wei-Min Wang[II] and Chao-Xi Jin[II]

[I] School of Chemical Engineering & Pharmaceutics, Henan University of Science and Technology, Luoyang 471003, Henan Province, P. R. China
[II] Luo Yang City Environmental Monitoring Station, Luoyang 471000, Henan Province, P. R. China

Received December 17, 2013, accepted March 31, 2014, available online May 05, 2014, CCDC no. 1267/4091



Abstract

$C_{11}H_{10}N_4S$, orthorhombic, *Pbca* (no. 61), a = 12.132(1) Å, b = 8.5791(8) Å, c = 21.292(2) Å, V = 2216.2 Å^3, Z = 8, $R_{gt}(F)$ = 0.0389, $wR_{ref}(F^2)$ = 0.1053, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.26×0.36×0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.68 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15224, 2061
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1751
$N(param)_{refined}$:	147
Programs:	SHELX [13]

Source of material

The title compound was obtained by the reaction of 1-(thiazolo[5,4-*b*]pyridin-2-yl)hydrazine (0.166 g, 1 mmol) and acetylacetone (0.1 g, 1 mmol) in methanol. Single crystals suitable for X-ray diffraction were obtained by crystallization of the title compound from methanol.

Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors U_{iso}(H) = 1.2 times U_{eq}(C, aromatic ring), and U_{iso}(H) = 1.5 times U_{eq}(C, methyl). The final refinement succeeded using geometrical restraints, with C–H = 0.93 Å (aromatic ring), and C–H = 0.96 Å (methyl).

Discussion

The thiazole ring has been found in many natural compounds that have useful biological activities [1-3]. Thiazoles and their derivatives have demonstrated to possess a wide range of physiology and pharmaceutical activities such as antitumor [4], anticonvulsant [5], antiviral [6], antibacterial [7], antimicrobial [8], and fungicidal activities [9]. On the other hand, the C–H···π interaction, which can be defined as the attraction between a C–H bond and a π system, has received much attention in recent years. Despite of being the weakest interaction among the hydrogen bonds, it has been found in a variety of substances to play important role in their physical, chemical, and biological properties [10, 11]. A view on the molecular structure of the title compound is given in the figure. The independent part of the unit cell contains one molecule of the title compound, which is constructed by the pyridine-thiazole moiety, and the dimethyl-pyrazol moiety. All non-hydrogen atoms of the title compound are almost planar with a maximum deviation of 0.7401(2) Å for C11. The C–N bond lengths are in the range of 1.293(2)-1.391(2) Å. The C1–S1 and C6–S1 bond lengths of 1.7455(18) Å and 1.7450(16) Å are intermediate between the double and single bonds. The shortening of the C–S bonds showing the partial double-bond character, which is in agreement with the literature data [12]. In the crystal structure, adjacent molecules are connected through weak intermolecular C–H···π interactions, leading to the formation of a 1D chain. The hydrogen atoms H10B and H11C interact with the neighbouring pyrazol ring (Cg1) and pyridine ring (Cg2), respectively, with d(H10B···Cg1) = 2.91 Å, d(C10···Cg1) = 3.4993(19) Å and C10–H10B···Cg1 = 120°; d(H11C···Cg2) = 2.92 Å, d(C11···Cg2) = 3.806(2) Å and C11–H11C···Cg2 = 154°.

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

Atom	Site	x	y	z	U_{iso}
H(3)	8*c*	0.7709	–0.3118	0.5010	0.062
H(4)	8*c*	0.6220	–0.4018	0.5595	0.066
H(5)	8*c*	0.4471	–0.3217	0.5360	0.065
H(8)	8*c*	0.9292	0.2068	0.2294	0.058
H(10A)	8*c*	0.7269	0.2409	0.1411	0.097
H(10B)	8*c*	0.7517	0.3957	0.1776	0.097
H(10C)	8*c*	0.6364	0.3150	0.1844	0.097
H(11A)	8*c*	1.0241	0.0304	0.3188	0.083
H(11B)	8*c*	0.9458	–0.1082	0.3367	0.083
H(11C)	8*c*	0.9526	0.0391	0.3801	0.083

* Correspondence author (e-mail: renyunlai2@126.com)

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*c*	0.5676(1)	–0.1351(2)	0.43274(8)	0.0343(8)	0.0472(9)	0.0425(9)	–0.0017(7)	0.0022(7)	–0.0016(7)
C(2)	8*c*	0.6777(1)	–0.1770(2)	0.44340(7)	0.0330(8)	0.0457(9)	0.0405(8)	–0.0043(7)	0.0007(6)	–0.0010(7)
C(3)	8*c*	0.6994(1)	–0.2798(2)	0.49195(8)	0.0384(9)	0.065(1)	0.052(1)	–0.0024(8)	–0.0035(8)	0.0103(9)
C(4)	8*c*	0.6108(2)	–0.3329(2)	0.52640(9)	0.051(1)	0.066(1)	0.049(1)	–0.0070(9)	0.0009(8)	0.0144(9)
C(5)	8*c*	0.5050(2)	–0.2836(2)	0.51174(9)	0.046(1)	0.067(1)	0.050(1)	–0.0089(9)	0.0081(8)	0.0064(9)
C(6)	8*c*	0.7023(1)	–0.0241(2)	0.36253(7)	0.0321(8)	0.0415(8)	0.0397(8)	–0.0019(6)	0.0011(6)	–0.0024(7)
C(7)	8*c*	0.8638(1)	0.0769(2)	0.30130(8)	0.0367(8)	0.0407(9)	0.0479(9)	–0.0025(7)	0.0068(7)	–0.0042(7)
C(8)	8*c*	0.8665(2)	0.1712(2)	0.24994(8)	0.049(1)	0.0462(9)	0.050(1)	–0.0055(8)	0.0125(8)	0.0000(8)
C(9)	8*c*	0.7563(2)	0.2048(2)	0.23369(8)	0.057(1)	0.0403(9)	0.0403(9)	–0.0017(7)	0.0043(8)	–0.0017(7)
C(10)	8*c*	0.7141(2)	0.2974(2)	0.17931(9)	0.082(1)	0.061(1)	0.052(1)	0.000(1)	–0.001(1)	0.012(1)
C(11)	8*c*	0.9546(2)	0.0030(2)	0.3374(1)	0.0375(9)	0.058(1)	0.071(1)	0.0005(8)	0.0062(9)	0.0071(9)
N(1)	8*c*	0.4806(1)	–0.1853(2)	0.46527(7)	0.0372(8)	0.065(1)	0.0544(9)	–0.0030(7)	0.0079(7)	0.0061(8)
N(2)	8*c*	0.7539(1)	–0.1114(2)	0.40236(6)	0.0326(7)	0.0501(8)	0.0437(7)	–0.0021(6)	0.0019(6)	0.0044(6)
N(3)	8*c*	0.7533(1)	0.0584(2)	0.31479(6)	0.0352(7)	0.0433(7)	0.0401(7)	–0.0010(6)	0.0016(6)	0.0016(6)
N(4)	8*c*	0.6867(1)	0.1377(2)	0.27308(6)	0.0451(8)	0.0479(8)	0.0416(8)	0.0021(6)	–0.0023(6)	0.0030(6)
S(1)	8*c*	0.55941(3)	–0.00755(5)	0.36903(2)	0.0321(3)	0.0591(3)	0.0527(3)	0.0035(2)	0.0014(2)	0.0103(2)

References

1. Gunawardana, G. P.; Kohmoto, S.; Gunasekera, S. P.; McConnell, O. J.; Koehn, F. E.: Dercitin, a new biologically active acridine alkaloid from a deep water marine sponge, Dercitus sp. J. Am. Chem. Soc. **110** (1998) 4856-4858.
2. Gunawardana, G. P.; Koehn, F. E.; Lee, A. Y.; Clardy, J.; He, H. Y.; Faulkner, D. J.: Pyridoacridine alkaloids from deep-water marine sponges of the family Pachastrellidae: structure revision of dercitin and related compounds and correlation with the kuanoniamines. J. Org. Chem. **57** (1992) 1523-1526.
3. Carroll, A. R.; Scheuer, P. J.: Kuanoniamines A, B, C, and D: pentacyclic alkaloids from a tunicate and its prosobranch mollusk predator Chelynotus spmpere. J. Org. Chem. 55 (1990) 4426-4431.
4. Andreani, A.; Leoni, A.; Locatelli, A.; Morigi, R.; Rambaldi, M.; Recanatini, M.; Garaliene, V.: Potential antitumor agent. Part 29[1]: synthesis and potential coanthracyclinic activity of imidazole[2,1-*b*]thiazole guanylhydrazones. Bioorg. Med. Chem. **8** (2000) 2359-2366.
5. Siddiqui, N.; Ahsan, W.: Triazole incorporated thiazoles as a new class of anticonvulsants: design, synthesis and in vivo screening. Eur. J. Med. Chem. **45** (2010) 1536-1543.
6. El-Sabbagh, O. J.; Baraka, M. M; Ibrahim, S. M.; Pannecouque, C.; Andrei, G.; Snoeck, R.; Balzarini, J.; Rashad, A. A.: Synthesis and antiviral activity of new pyrazole and thiazole derivatives. Eur. J. Med. Chem. **44** (2009) 3746-3753.
7. Bharti, S. K.; Nath, G.; Tilak, R.; Singh, S. K.: Synthesis, anti-bacterial and anti-fungal activities of some novel Schiff bases containing 2,4-disubstituted thiazole ring. Eur. J. Med. Chem. **45** (2010) 651-660.
8. Karegoudar, P.; Karthikeyan, M. S.; Prasad, D. J.; Mahalinga, M.; Holla, B. S.; Kumari, N. S.: Synthesis of some 2,4-disubstituted thizaoles as possible antimicrobial agents. Eru. J. Med. Chem. **43** (2008) 261-267.
9. Weng. J. Q.; Tan, C. X.; Liu, X. H.: Synthesis and fungicidal activity of hydrazones containing 4-methylbenzo[*d*]thiazole moiety. J. Pestic. Sic. **37** (2012) 164-168.
10. Nishio, M.; Hirota, M.; Umezawa, Y.: The C–H···π interaction. Wiley-VCH, New York, 1998.
11. Umezawa, Y.; Tsuboyama, S; Takahashi, H.; Uzawa, J.; Nishio, M.: C–H···π interaction in the conformation of organic compounds. A database study. **55** (1999) 10047-10056.
12. Lawrence, H. R.; Li, Z.; Yip, M. L. R.; Sung, S. S.; Lawrence, N. J.; McLaughlin, M. L.; McManus, G. J.; Zaworotko, M. J.; Sebti, S. M.; Chen, J.; Guida, W. C.: Indentification of a disruptor of the MDM2-p53 protein-protein interaction facilitated by high-throughput in silico docking. Bioorg. Med. Chem. Lett. **19** (2009) 3756-3759.
13. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.