Crystal structure of 1-(4,6-dimethylpyrimidin-2-yl)benzimidazole, $C_{13}H_{12}N_4$

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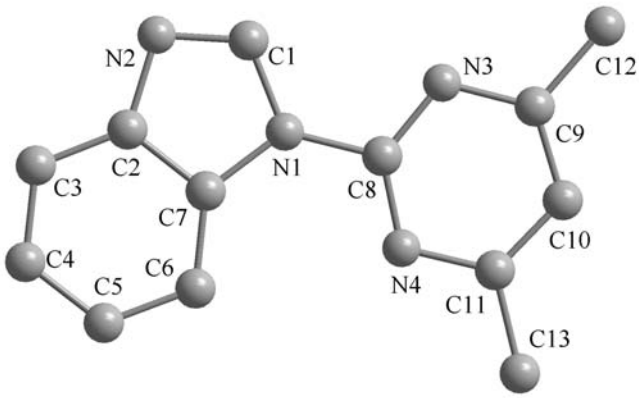

Abstract

$C_{13}H_{12}N_4$, monoclinic, $P12_1/c1$ (no. 14), a = 8.682(1) Å, b = 17.542(3) Å, c = 7.571(1) Å, β = 90.649(2)°, V = 1153.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.043, $wR_{ref}(F^2)$ = 0.111, T = 296 K.

Source of material

Benzimidazole (3 mmol) and NaH (9 mmol) were dissolved in dry dioxane (25 ml) and the solution was stirred for 2 h at room temperature, then 4,6-dimethyliodopyrimidine (3 mmol) was added. The resultant solution was heated to reflux for 6 h, removal of solvent resulted in a white powder that was recrystallized from dichloromethane/petroleum ether solution at room temperature to give the desired crystals suitable for single crystal X-ray diffraction.

Discussion

Benzimidazole is an interesting heterocyclic ligand with nitrogen as the donor atom because it is a component of biologically important molecules [1]. Benzimidazole derivatives and their metal complexes have been extensively investigated recently [2,3]. Additionally, benzimidazolium salts are widely used in preparation of N-heterocyclic carbene ligands [4,5]. Among them, only few pyrimidine-functionalized NHC ligands have been reported [6,7].

The pyrimidine ring and benzimidazole ring of the title molecule are approximately coplanar with a dihedral angle of 2.5°. All the bond distances and angles are within normal ranges. The H atoms of the methyl groups in the pyrimidine ring are disordered over two positions; their site-occupation factors were fixed at 0.5. There exist two types of intermolecular C–H···N hydrogen bonds (the distances are 2.748 Å and 2.645 Å), which construct the chain structure. In addition, there are two types of intermolecular π-π stacking interactions. One π-π stacking interaction is between the pyrimidine rings of the two chains (the interplane distance is 3.414 Å) [8]. The other π-π stacking interaction is pyrimidine ring and benzimidazole ring (the interplane distance 3.593 Å), which link the molecules into a layers.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.08 × 0.19 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.82 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8476, 2143
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1364
$N(param)_{refined}$:	155
Programs:	SHELXS-97, SHELXL-97, SHELXTL [9]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	4*e*		–0.1120	0.3435	0.5880	0.070
H(3)	4*e*		0.3388	0.2192	0.4031	0.080
H(4)	4*e*		0.5788	0.2723	0.4493	0.089
H(5)	4*e*		0.6055	0.3900	0.5816	0.088
H(6)	4*e*		0.3915	0.4585	0.6722	0.073
H(10)	4*e*		–0.0903	0.6391	0.9169	0.063
H(12A)	4*e*	0.50	–0.3814	0.5011	0.8087	0.102
H(12B)	4*e*	0.50	–0.3689	0.5899	0.7908	0.102
H(12C)	4*e*	0.50	–0.3451	0.5511	0.9757	0.102
H(12D)	4*e*	0.50	–0.3489	0.5937	0.9081	0.102
H(12E)	4*e*	0.50	–0.3614	0.5048	0.9260	0.102
H(12F)	4*e*	0.50	–0.3851	0.5436	0.7411	0.102
H(13A)	4*e*	0.50	0.3163	0.6225	0.8476	0.112
H(13B)	4*e*	0.50	0.2153	0.6485	1.0070	0.112
H(13C)	4*e*	0.50	0.1955	0.6881	0.8225	0.112
H(13D)	4*e*	0.50	0.1685	0.6836	0.9372	0.112
H(13E)	4*e*	0.50	0.2694	0.6575	0.7778	0.112
H(13F)	4*e*	0.50	0.2892	0.6180	0.9623	0.112

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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)	4*e*	–0.0056(2)	0.3424(1)	0.5758(3)	0.057(1)	0.053(1)	0.066(1)	–0.009(1)	0.000(1)	–0.000(1)
C(2)	4*e*	0.2208(2)	0.3065(1)	0.5139(2)	0.065(1)	0.044(1)	0.049(1)	0.004(1)	0.0009(9)	0.0033(9)
C(3)	4*e*	0.3491(3)	0.2665(1)	0.4577(3)	0.089(2)	0.051(1)	0.061(1)	0.012(1)	0.007(1)	–0.002(1)
C(4)	4*e*	0.4913(3)	0.2985(1)	0.4848(3)	0.072(2)	0.074(2)	0.078(2)	0.026(1)	0.013(1)	0.003(1)
C(5)	4*e*	0.5075(2)	0.3697(1)	0.5648(3)	0.055(1)	0.079(2)	0.086(2)	0.010(1)	0.003(1)	–0.004(1)
C(6)	4*e*	0.3809(2)	0.4108(1)	0.6198(3)	0.052(1)	0.060(1)	0.070(1)	0.004(1)	0.000(1)	–0.005(1)
C(7)	4*e*	0.2381(2)	0.3779(1)	0.5934(2)	0.049(1)	0.047(1)	0.046(1)	0.0042(9)	0.0023(8)	0.0033(9)
C(8)	4*e*	0.0378(2)	0.46944(9)	0.7121(2)	0.048(1)	0.043(1)	0.043(1)	–0.0016(9)	0.0001(8)	0.0042(8)
C(9)	4*e*	–0.1603(2)	0.5386(1)	0.8165(2)	0.048(1)	0.055(1)	0.047(1)	0.0044(9)	–0.0005(8)	0.005(1)
C(10)	4*e*	–0.0567(2)	0.5945(1)	0.8635(2)	0.054(1)	0.048(1)	0.055(1)	0.0050(9)	0.0042(9)	0.0009(9)
C(11)	4*e*	0.0970(2)	0.5836(1)	0.8310(2)	0.054(1)	0.043(1)	0.053(1)	–0.0021(9)	0.0024(9)	0.0033(9)
C(12)	4*e*	–0.3290(2)	0.5458(1)	0.8510(3)	0.051(1)	0.078(2)	0.075(2)	0.007(1)	0.003(1)	–0.004(1)
C(13)	4*e*	0.2167(2)	0.6408(1)	0.8815(3)	0.067(1)	0.058(1)	0.098(2)	–0.011(1)	0.007(1)	–0.012(1)
N(1)	4*e*	0.0885(2)	0.40104(8)	0.6325(2)	0.0477(9)	0.0423(9)	0.0540(9)	–0.0022(7)	0.0004(7)	–0.0020(7)
N(2)	4*e*	0.0658(2)	0.28553(9)	0.5043(2)	0.076(1)	0.048(1)	0.068(1)	–0.0055(9)	0.0041(9)	–0.0038(8)
N(3)	4*e*	–0.1133(2)	0.47413(8)	0.7377(2)	0.0435(9)	0.054(1)	0.051(1)	–0.0003(7)	–0.0007(7)	0.0014(8)
N(4)	4*e*	0.1469(2)	0.51916(8)	0.7526(2)	0.0485(9)	0.0459(9)	0.056(1)	–0.0027(7)	0.0029(7)	–0.0002(8)

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References

1. Reedijk, J.: Comprehensive Coordination Chemistry. Vol. 2. Pergamon, Oxford, 1987.
2. Preston, P. N.: Synthesis, reaction, and spectroscopic properties of benzimidazoles. Chem. Rev. **74** (1974) 279-314.
3. Bouwman, E.; Driessen, W. L.; Reedijk, J.: Model systems for type I copper coordination compounds with thioether and azole-containing ligands. Coord. Chem. Rev. **104** (1990) 143-172.
4. Kantchev, E. A. B.; O'Brien, C. J.; Organ, M. G.: Palladium Complexes of N-Heterocyclic Carbenes as Catalysts for Cross-Coupling Reactions - A Synthetic Chemist's Perspective. Angew. Chem., Int. Ed. **46** (2007) 2768-2813.
5. Hahn, F. E.; Jahnke, M. C.: Heterocyclic Carbenes: Synthesis and Coordination Chemistry. Angew. Chem., Int. Ed. **47** (2008) 3122-3172.
6. Zhang, X. M.; Liu, B.; Liu, A. L; Xie, W. L.; Chen, W. Z.: Steric Bulkiness-Dependent Dtructural Diversity in Nickel Complexes of N-Heterocyclic Carbenes: Synthesis and Structural Characterization of Tatra-, Penta-, and Hexa-coordinated Nickel Complexes. Organometallics **28** (2009) 1336-1349.
7. Liu, B.; Xia, Q. Q.; Chen, W. Z.: Direct Synthesis of Iron, Cobalt, Nickel, and Copper Complexes of N-Heterocyclic Carbenes by Using Commercially Available Metal Powders. Angew. Chem., Int. Ed. **48** (2009) 5513-5516.
8. Xu, C.; Wang, Z. Q.; Fu, W. J.; Lou, X. H.; Li, Y. F.; Cen, F. F.; Ma, H. J.; Ji, B. M.: Synthesis and Structural Characterization of Monophosphine-Cyclopalladated Ferrocenylpyrimidine Complexes and Reusable Catalytic System for Amination of Hindered Aryl Chlorides in PEG-400. Organometallics **28** (2009) 1909-1916.
9. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.