Crystal structure of 2-(2-amino-5-chlorophenyl)-1*H*-benzimidazole, $C_{13}H_{10}ClN_3$

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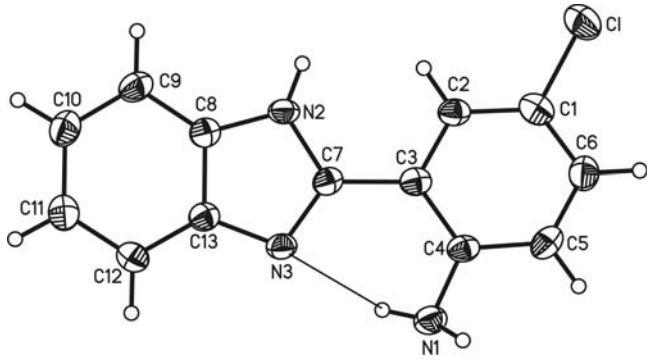

Abstract

$C_{13}H_{10}ClN_3$, orthorhombic, $Pna2_1$ (no. 33), a = 8.685(2) Å, b = 6.648(1) Å, c = 19.264(4) Å, V = 1112.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.149, T = 296 K.

Source of material

A mixture of 1,2-phenylenediamine (1.08 g, 0.01 mol) and 2-amino-5-chlorobenzoic acid (2.14 g, 0.012 mol) was stirred in the sufficient amount of polyphosphate for 5 - 6 hours at 473 - 493 K to afford the title compound (1.51 g, yield 62 %). Single crystals were obtained by recrystallization from acetonitrile at room temperature over a period of one week (mp. 466 - 468 K).

Experimental details

All H atoms were located from the difference Fourier maps and treated as riding with d(C—H) = 0.93 Å, U_{iso}(H) = 1.2 U_{eq}(C); d(N_{amine}—H) = 0.89 Å and d($N_{imidazole}$—H) = 0.86 Å; U_{iso}(H) = 1.5 U_{eq}(N). The Flack parameter x = 0.15(12) confirms the determined absolute structure.

Discussion

Benzimidazole and their derivatives are known to exhibit a wide variety of pharmacological properties. Benzimidazole is an important pharmacophore and a privileged structure in medicinal chemistry encompassing a diverse range of biological activities [1]. Series of DNA-interactive benzimidazole analogues, such as Hoechst 33258 [2], were also prepared to explore the potential for anticancer activity mediated for certain of drugs via bio-reductive activation by endogenous nicotinamide adenine dinucleotide or nicotinamide adenine dinucleotide 2′-phosphate reduced tetrasodium salt [3]. Our research group has focused on the synthesis of benzimidazole analogues.

In the crystal structure, the main moiety is twisted with the dihedral angle of benzimidazole and benzene being 20.80(1)°. Compared with the other reported phenylbenzimidazole system [4-6], it adapts twisted configuration in a smaller degree. It can be explained by an important intra-molecular hydrogen bond between the amino group at the *para*-position to the Cl atom and the imidazole N atom (N1–H···N3). The distance of benzimidazole planes of the adjacent molecules is 3.516(3) Å showing much stronger $\pi\cdots\pi$ interaction along [100] compared with the corresponding distances 3.994(2) Å and 3.734(2) Å described in the similar benzimidazole analogues [7,8]. There are also intermolecular hydrogen bonds N1–H···N3 ($\frac{1}{2}+x,\frac{5}{2}-y,z$) and N2–H···N1 ($x,-1+y,z$) stabilizing the whole crystal structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.10 × 0.10 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.21 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	50.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2002, 2002
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1668
$N(param)_{refined}$:	154
Programs:	SHELXS-97, SHELXL-97 [9]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*a*	0.5790	1.3518	0.3608	0.074
H(1B)	4*a*	0.4632	1.2212	0.3302	0.074
H(2A)	4*a*	0.4588	0.6712	0.4687	0.050
H(2B)	4*a*	0.3723	0.5741	0.3658	0.048
H(5A)	4*a*	0.6762	1.3040	0.4729	0.059
H(6A)	4*a*	0.7407	1.0705	0.5571	0.060
H(9A)	4*a*	0.2068	0.3785	0.2581	0.059
H(10A)	4*a*	0.0529	0.4590	0.1633	0.066
H(11A)	4*a*	–0.0030	0.7890	0.1371	0.062
H(12A)	4*a*	0.0981	1.0490	0.2019	0.061

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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}
Cl	4*a*	0.6545(2)	0.6743(2)	0.58275(7)	0.0719(7)	0.0682(8)	0.0559(6)	0.0076(6)	–0.0088(6)	0.0127(6)
N(1)	4*a*	0.4946(4)	1.2799(5)	0.3693(2)	0.045(2)	0.037(2)	0.066(2)	–0.004(2)	–0.001(2)	0.004(2)
C(1)	4*a*	0.6052(5)	0.8505(6)	0.5196(2)	0.042(2)	0.054(2)	0.044(2)	0.010(2)	0.009(2)	0.006(2)
C(2)	4*a*	0.5021(5)	0.7992(5)	0.4688(2)	0.035(2)	0.035(2)	0.054(2)	0.001(2)	0.005(2)	0.004(2)
N(2)	4*a*	0.3357(4)	0.6763(5)	0.3440(2)	0.040(2)	0.029(2)	0.050(2)	0.001(1)	0.000(2)	0.007(1)
N(3)	4*a*	0.2857(4)	0.9963(4)	0.3166(2)	0.039(2)	0.030(2)	0.053(2)	0.004(1)	–0.001(2)	–0.001(2)
C(3)	4*a*	0.4612(4)	0.9371(5)	0.4172(2)	0.028(2)	0.036(2)	0.050(2)	0.003(2)	0.009(2)	0.001(2)
C(4)	4*a*	0.5286(4)	1.1314(5)	0.4183(2)	0.033(2)	0.033(2)	0.054(2)	0.004(2)	0.008(2)	–0.001(2)
C(5)	4*a*	0.6321(5)	1.1766(6)	0.4715(2)	0.048(2)	0.043(2)	0.056(2)	–0.006(2)	0.006(2)	–0.014(2)
C(6)	4*a*	0.6713(5)	1.0375(7)	0.5221(2)	0.045(2)	0.054(3)	0.051(2)	–0.002(2)	–0.005(2)	–0.005(2)
C(7)	4*a*	0.3603(4)	0.8740(5)	0.3601(2)	0.027(2)	0.033(2)	0.052(2)	–0.002(2)	0.006(2)	–0.001(2)
C(8)	4*a*	0.2416(4)	0.6706(6)	0.2866(2)	0.030(2)	0.040(2)	0.049(2)	–0.001(2)	0.007(2)	0.001(2)
C(9)	4*a*	0.1845(4)	0.5116(7)	0.2472(2)	0.048(2)	0.038(2)	0.062(3)	–0.006(2)	0.005(2)	–0.001(2)
C(10)	4*a*	0.0931(5)	0.5610(7)	0.1909(2)	0.048(2)	0.055(3)	0.062(3)	–0.008(2)	–0.003(2)	–0.008(2)
C(11)	4*a*	0.0603(5)	0.7605(7)	0.1748(2)	0.047(2)	0.060(3)	0.049(2)	0.001(2)	–0.003(2)	–0.005(2)
C(12)	4*a*	0.1194(5)	0.9161(7)	0.2134(2)	0.047(2)	0.046(2)	0.059(3)	0.008(2)	–0.007(2)	0.003(2)
C(13)	4*a*	0.2114(4)	0.8702(6)	0.2699(2)	0.035(2)	0.038(2)	0.048(2)	0.002(2)	0.004(2)	–0.001(2)

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