Synthesis and Structural Characterization of 2-(2-Aminophenyl) Benz imidazole

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Abstract. A Benz imidazole derivative, 2-(2-aminophenyl) Benz imidazole, was synthesized by simple one-pot synthesis method. Its structure was characterized by X-ray single-crystal structural analysis. The results of crystal analysis are $C_{13}H_{11}N_3$, M_r = 209.25, orthorhombic, space group *Pbca*, a = 7.7679 (16) Å, b = 11.855(2) Å, c = 22.552(5) Å, $\alpha = \beta = \gamma$ = 90º, V= 2076.7(7) Å^3, Z = 8, Dc = 1.339 g·cm^{-3}, μ = 0.083 mm^{-1}, F (000) = 880, and final R_1 = 0.0365, ωR_2 = 0.0947. The 2-(2-aminophenyl) Benz imidazole molecule is co-planar. The title compound molecules form 1D chained structure and 2D layered structure by the interaction of intermolecular N-H.N hydrogen bonds.

1. Introduction

Benz imidazole derivatives are a class of important heterocyclic compounds, they have many physiological activities such as antiviral activity, and antioxidant, anticancer activity and ant proliferative activity [1-4] .So the study on the synthesis and structure of Benz imidazole derivatives have important scientific significance. 2-(2-aminophenyl) Benz imidazole has been synthesized and structural characterized by Das's study group [5]. However, they use the method of reducing 2-(o-nitro phenyl) Benz imidazole to 2-(2-aminophenyl) Benz imidazole in the atmosphere of hydrogen gas with Pd/C as catalyst. In this paper, 2-(2-aminophenyl) Benz imidazole was synthesized by a simple one-pot method using 2-aminobenzaldehyde and 1, 2-diaminobenzene as raw materials. The structure of 2-(2-aminophenyl) Benz imidazole has been structural characterized.

2. Experimental Section

2.1. Materials and Instrumentation

2-Aminobenzaldehyde and 1, 2-diaminobenzene were purchased from Xiya reagent Company. The single-crystal diffraction data were collected on a Bruker Smart-1000 CCD diffractometer.

2.2. Synthesis of 2-(2-Aminophenyl) Benz imidazole

1.0 mmol 2-aminobenzaldehyde (0.1211 g) and 1.0 mmol 1, 2-diaminobenzene (0.1081 g) was dissolved in 10 mL ethanol with stirring at room temperature. The mixture was stirred for 7 h at 65 °C, and then was cooled to room temperature. The result solution was filtered and left at room temperature. The needle-shaped crystals of 2-(2-aminophenyl) Benz imidazole were obtained after 15 days.

2.3. Crystal Data and Structure Determination

The structure of 2-(2-aminophenyl) Benz imidazole was solved by direct method using SHELXL-97 [6] and refined on F^2 by full-matrix least-squares with SHELXTL-97 [7]. The crystal data of 2-(2-aminophenyl) Benz imidazole was listed in Table 1.

Table 1. Crystal data for 2-(2-aminophenyl) Benz imidazole

Formula	$C_{13}H_{11}N_3$
Formula weight	209.25
Crystal system	orthorhombic
Space group	*Pbca*
a [Å]	7.7679(16)
b [Å]	11.855(2)
c [Å]	22.552(5)
$\alpha=\beta=\gamma$ [º]	90
Crystal size	0.21×0.20×0.19 mm
Exptl absorpt correction type	multi-scan
Z	8
$F(000)$	880
Temperature [K]	293(2)
V [Å^3]	2076.7(7)
Calculated density [g·cm^{-3}]	1.339
μ [mm^{-1}]	0.083
S	1.076
Limiting indices	$-9 \le h \le 8$, $-14 \le k \le 14$, $-26 \le l \le 26$
Reflections collected	14866
Unique reflections	1831
Parameters	145
Restraints	0
R_{int}	0.0289
R_1, wR_2 [all data]	0.0418, 0.1007
R_1, wR_2 [$I>2\sigma(I)$]	0.0365, 0.0947
Largest diff.peak and hole [e·Å^{-3}]	0.255, -0.146
Molecular graphics	DIAMOND

3. Results and Discussion

3.1. Structural Description of 2-(2-Aminophenyl) Benz imidazole

In the title compound, the bond distances of C7-N1, C7-N2 and C13-N3 are 1.3194(16), 1.3710(16) and 1.3936(16) Å, respectively, indicating that the C7-N1 bond is double bond. The dihedral angle between the two benzene rings (C1-C2-C3-C4-C5-C6 and C8-C9-C10-C11-C12-C13) is 7.2°, showing that the 2-(2-aminophenyl) Benz imidazole molecule is co-planar. The title compound molecules form 1D chained structure and 2D layered structure by the interaction of intermolecular N-H...N hydrogen bonds (N2-H2B…N3: 0.860 Å, 2.241 Å, 3.083 Å, 166.27°, symmetry code: -*x*+1/2, *y*-1/2, *z*; N3-H3D-N1: 0.969 Å, 1.974 Å, 2.739 Å, 134.22°). The molecular structure of 2-(2-aminophenyl) Benz imidazole is given in Figure 1. The 1D chained structure and 2D layered structure is shown in Figure 2. The bond lengths and bond angles are listed in Table 2.

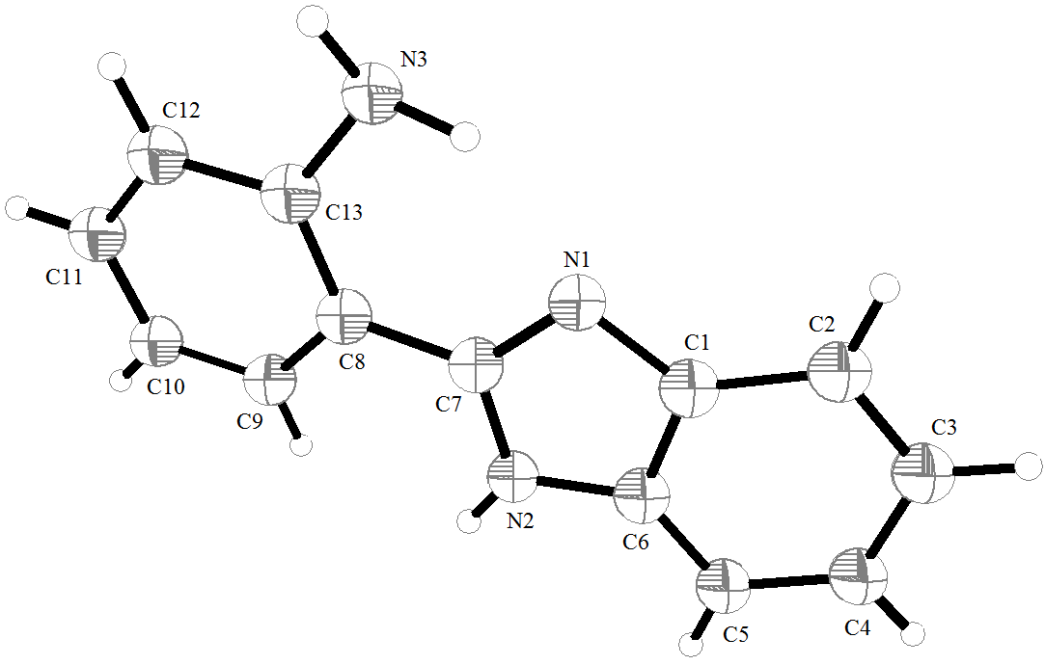

Figure 1. The molecular structure of 2-(2-aminophenyl) Benz imidazole

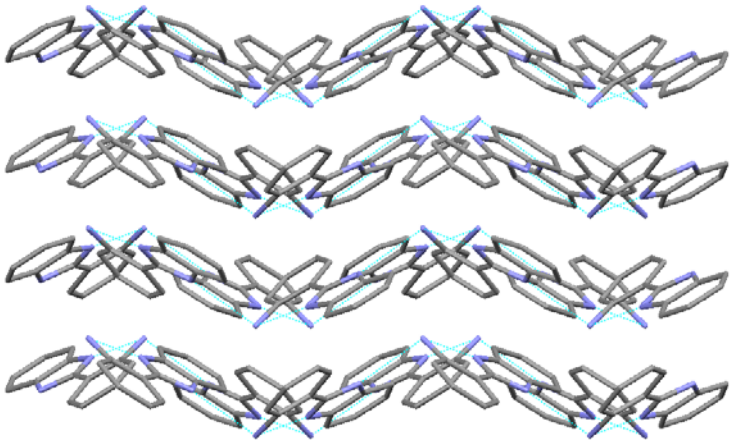

Figure 2. 1D and 2D structure of 2-(2-aminophenyl) Benz imidazole

Table 2. Selected bond distances and bond angles for 2-(2-aminophenyl) Benz imidazole

Bond	Distance(Å)	Bond	Distance(Å)
C7-N1	1.3194(16)	C1-C2	1.3979(18)
C1-N1	1.3864(16)	C2-C3	1.374(2)
C7-N2	1.3710(16)	C3-C4	1.400(2)
C6-N2	1.3737(16)	C4-C5	1.372(2)
C13-N3	1.3936(16)	C5-C6	1.3902(18)
C1-C6	1.3972(17)	C7-C8	1.4642(17)
C8-C9	1.3966(18)	C8-C13	1.4112(17)
Angle	(°)	Angle	(°)
C7-N1-C1	105.47(10)	C7-N2-C6	107.35(10)
N1-C1-C6	109.77(11)	N1-C1-C2	130.11(12)
C6-C1-C2	120.08(12)	C3-C2-C1	117.61(13)
C2-C3-C4	121.50(13)	C5-C6-N2	132.27(12)
C1-C6-N2	105.39(10)	N1-C7-N2	112.02(11)
N1-C7-C8	125.36(11)	N2-C7-C8	122.62(10)
C12-C13-N3	119.17(11)	C8-C13-N3	122.13(11)
C10-C11-C12	120.14(13)	C11-C12-C13	121.77(13)
C12-C13-C8	118.63(11)	C3-C4-C5	121.82(13)
C7-C8-C9	120.08(11)		

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