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Synthesis and Crystal Structure of 1-(2-(1*H*-Benzoimidazol-1-yl)ethyl)-1*H*-benzoimidazol-3-ium Chloride Hydrate

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The crystal structure of 1-(2-(1*H*-benzoimidazol-1-yl)ethyl)-1*H*-benzoimidazol-3-ium chloride hydrate has been determined by single crystal X-ray diffraction method. The crystal belongs to monoclinic system, space group C2/c with unit cell constants a = 17.093 (3), b = 7.764 (1), c = 13.952 (3) Å, V = 1652.7 (5) Å^3, Z = 4, Dc = 1.346 g/cm^3, μ = 0.246 mm^{-1}, F (000) = 704, R and wR are 0.0718 and 0.1418, respectively for 1561 unique reflections with 1498 observed reflections [I > 2σ(I)]. The crystal packing is stabilized by C-H…π and π-π interactions.

Key Words: Synthesis, Crystal structure, Benzimidazole derivatives.

INTRODUCTION

Benzimidazole derivatives, a kind of important heterocyclic compounds, have been extensively applied, better properties in the fields of composite materials, electronic chemistry, metal anticorrosive agents, photosensitive materials as well as in biology and medicine[1-3]. *Bis*(1-benzimidazole)ethane is the important one. Our target product, *bis*(1-benzimidazole)ethane, was synthesized by using benzimidazole and glycol in THF. It has good anticancer function and its crystal structure is presented here.

EXPERIMENTAL

Glycol (0.025 mol, 1.55 g) was added dropwise to a solution of tosyl chloride (0.05 mol, 10.0 g) in THF (45 mL) at 281 K. The mixture was stirred for 2 h and adjust pH to 7 after 2 h. Then benzimidazole (0.05 mol, 5.85 g) was added dropwise to the solution. The solution was stirred and refluxed for 6 h. The resulting white solid was filtered and washed with water. Single crystal suitable for X-ray diffraction study were obtained by slow evaporation of the product solution.

All reagents were obtained from commercial suppliers and were used without further purification.

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Crystal data and structure determination: A colourless single crystal with approximate dimension of 0.42 mm × 0.35 mm × 0.10 mm was mounted on glass fibre in a random orientation. The data were collected by Bruker Smart 1000 CCD diffractometer with graphite minochromated MoK_α radiation (λ = 0,71073 Å) using ω scan mode in the range of $2.67 \leq \theta \leq 25.64^\circ$ at temperature 293 (±2) K. A total of 4323 reflections were collected with 1561 unique ones (R_{int} = 0.016), of which 1498 reflections with $I > 2\sigma(I)$ were considered to be observed and used in the succeeding refinements. Intensity data were corrected for Lp factors and empirical absorption. The structure was solved by direct methods and expanded by using Fourier differential techniques with SHELXL-97[4]. All non-hydrogen atoms were located with successive difference Fourier syntheses. The structure was refined by full-matrix least-squares method on F^2 with anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were added according to the theoretical models. Full matrix least-squares refinement gave the final R = 0.0718 and wR = 0.1423, $w = 1/[\sigma^2(Fo)^2 + (0.0000P)^2 + 6.2407P]$ where $P = (Fo^2 + 2Fc^2)/3$.

RESULTS AND DISCUSSION

The final atomic parameters and equivalent isotropic thermal parameters for the non-hydrogen atoms are given in Table-1. Selected bond lengths and bond angles are listed in Tables 2 and 3, respectively. Fig. 1 show the molecular structure of the compound. Packing diagram of the title compound in a unit cell is shown in Fig. 2.

TABLE-1
ATOMIC COORDINATES AND EQUIVALENT ISOTROPIC DISPLACEMENT PARAMETERS ($Å^2 \times 10^3$)

Atom	x	y	z	Ueq
N1	0.7839(2)	1.0235(4)	0.9908(2)	0.0441(10)
N2	0.8745(2)	0.8415(4)	0.9717(2)	0.0512(11)
C1	0.8672(3)	0.9705(5)	1.0283(3)	0.0488(11)
C2	0.7331(2)	0.9203(4)	0.9034(3)	0.0396(10)
C3	0.6442(3)	0.9187(5)	0.8353(3)	0.0502(12)
C4	0.6157(3)	0.7957(6)	0.7553(3)	0.0606(16)
C5	0.6733(3)	0.6804(6)	0.7443(3)	0.0641(16)
C6	0.7614(3)	0.6817(5)	0.8119(3)	0.0555(14)
C7	0.7905(3)	0.8056(4)	0.8923(3)	0.0430(11)
C8	0.7543(3)	1.1693(4)	1.0329(3)	0.0484(14)
O1W	1.0133(3)	0.6649(5)	0.9745(3)	0.0703(12)
Cl	1	0.7838(2)	3/4	0.0703(6)

In the molecule of the title compound (Fig. 1), the bond lengths and angles are within normal ranges[5]. The benzotriazole system is essentially planar, with a dihedral angle of 1.8(2)° between the N1-N3/C1/C2 triazole ring and C1-C6 benzene ring. The whole molecule is non-planar. The mean benzotriazole plane makes dihedral angles of 39.10(1)°-20.55(2)° with the C10-C15-C17-C22 benzene rings, respectively. The dihedral angle between the latter two aromatic rings is 53.25(2)°.

TABLE-2
SELECTED BOND LENGTHS (Å)

Bond	Distance (Å)	Bond	Distance (Å)
O1W-H1BW	0.68(11)	C2-C7	1.384(6)
O1W-H1AW	0.79(5)	C2-C3	1.382(6)
N1-C2	1.389(5)	C3-C4	1.380(6)
N1-C1	1.341(6)	C4-C5	1.389(7)
N1-C8	1.466(5)	C5-C6	1.370(7)
N2-C7	1.390(6)	C6-C7	1.389(5)
N2-C1	1.316(5)	C8-C8A	1.521(5)

TABLE-3
SELECTED BOND ANGLES (°)

Angle	(°)	Angle	(°)
H1AW-O1W-H1BW	113(9)	C3-C2-C7	122.2(3)
C1-N1-C2	107.4(3)	C2-C3-C4	116.1(4)
C1-N1-C8	125.3(3)	C3-C4-C5	121.7(4)
C2-N1-C8	127.2(3)	C4-C5-C6	122.2(4)
C1-N2-C7	106.7(4)	C5-C6-C7	116.3(4)
C1-N2-H2A	126.62	N2-C7-C2	108.1(3)
C7-N2-H2A	126.67	N2-C7-C6	130.4(4)
N1-C1-N2	111.8(4)	C2-C7-C6	121.5(4)
N1-C2-C3	131.9(3)	N1-C8-C8A	110.6(3)
N1-C2-C7	106.0(3)	–	–

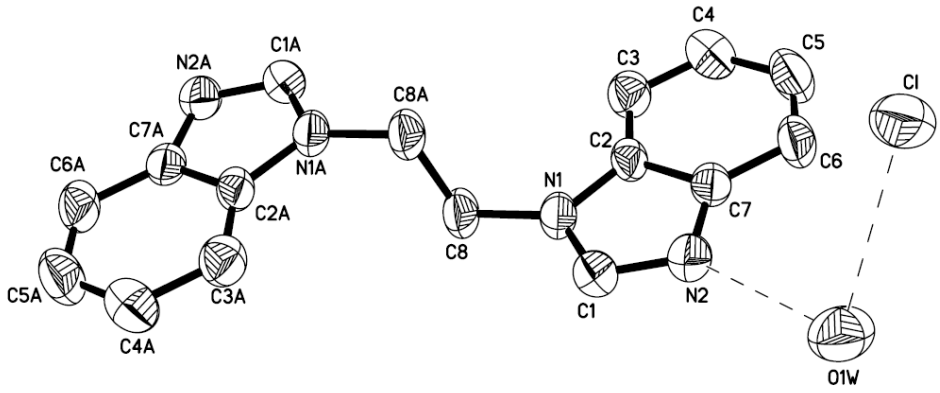

Fig. 1. Molecular structure of the title compound with the atomic numbering Scheme

In the crystal structure, the short distance Cg1…Cg3^{i} [symmetry code: (i) 1/2-x, 1-y, 1/2 + z], where Cg1 and Cg3 denote the centroids the N1-N3/C1/C2 triazole ring and C10-C15 benzene ring, respectively, indicates the π-π interactions. The crystal packing is further stabilized by van der Waals forces.

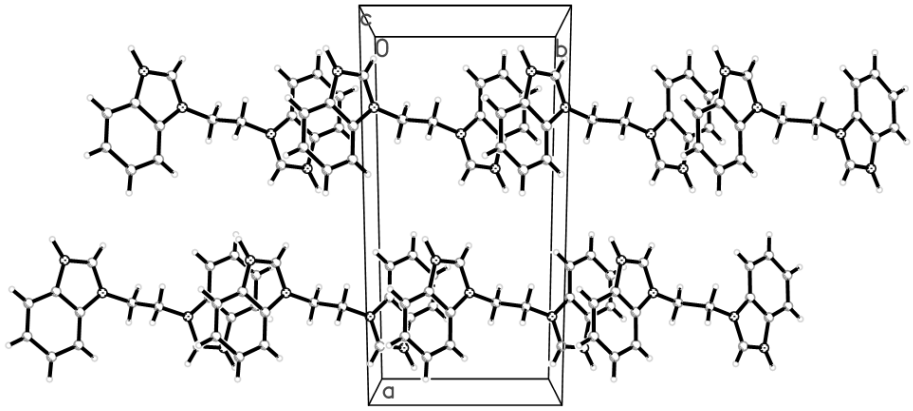

Fig. 2. A view of the crystal packing for the title compound

The molecular of the title compound, (Fig. 1), has a crystallographically imposed center of symmetry at the midpoint of the C8-C8A^{i} bond [symmetry code: (i): 3/2-x, 5/2-y, 2-z] with the occupancy value of H atom on N2 is 0.5 because it is disorder. All bond lengths and angles are within normal ranges[5]. The unit of the molecular also contains one water molecule and one chloride in order to balance the charge. In the crystal structure, molecules are also stabilized by C5-H5...Cg1 interactions. The short distances Cg1...Cg1^{ii} (3.766 Å) and Cg1...Cg2^{ii} (3.566 Å) [symmetry code: (ii) 3/2-x, 3/2-y, 2-z], where Cg1 and Cg2 denote the centroids of N1/N2/C1/C2/C7 imidazole ring and C2-C7 benzene ring, respectively, indicate π-π interactions.

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