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Crystal Structure of 1-(Propa-1,2-dienyl)-1*H*-benzo(*d*)imidazole-2-carbaldehyde

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The structure of 1-(propa-1,2-dienyl)-1*H*-benzo[*d*]imidazole-2-carbaldehyde was determine by X-ray crystallography. The compound crystallized in a monoclinic system, and was characterized as: $P2_1/n$, a = 3.9124(8), b = 15.082(3), c = 15.407(3)Å, β = 91.784(4)°, Z = 4, V = 908.7(3)Å^3. The crystal structure was solved by direct methods and refined by full-matrix least-squares on F^2 to final values of $R1$ = 0.045 and $wR2$ = 0.133.

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Imidazole derivatives are used as p38 MAP kinase inhibitors.[1] These derivatives are used as antimicrobial and antituberculosis agents.[2] The benzimidazole nucleus is found in a variety of naturally occurring compounds, such as vitamin B_{12} and its derivatives. The benzimidazole group possess great thermal stability, and has been used as part of the backbone in high performance, high-temperature polymers.[3] The benzimidazole moiety is known to be an essential structural component of telmisartan for PPAR gamma activation.[4] In continuation of our work on the crystal structure analysis of imidazole derivatives, we have undertaken a single-crystal X-ray diffraction study for the title compound, present the results here.

The title compound was prepared by a mixure of 1*H*-benzo[*d*]imidazole carbaldehyde (20 mmol) and propargyl bromide (35 mmol) refluxed tetrahydrofuran under an acidic condition for about 24 h, and the solvent was removed under reduced pressure. The crude product was subjected to column chromatography using a hexane and ethylacetate mixture (3:2) to obtain the title compound (Fig. 1). Single crystals suitable for X-ray structure analysis could be obtained by crystallization from a chloroform and methanol (1:1) solution.

Three-dimensional intensity data were collected by using a Bruker SMART APEX CCD area detector diffractometer. The structure was solved by direct methods using SHELXS97 software. A full-matrix least-squares refinement of the non-hydrogen atoms with isotropic temperature factors was carried out using SHELXL97. Subsequent cycles of refinement yielded a final R-factor of 0.045.

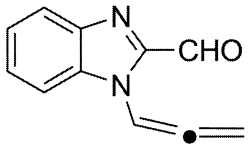

Fig. 1 Chemical diagram of the title compound.

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The crystal and experimental data are given in Table 1. An ORTEP view of the molecule with atomic labeling (thermal ellipsoids are drawn at 50% probability) is shown in Fig. 2. The atomic coordinates for non-hydrogen atoms are given in Table 2.

All of the C–C, C–N and C=N bond lengths of the benzimidazole moiety agree with the related reported structure of the benzimidazole moiety.[5] The double-bond distance in dienyl group is comparable to the literature value of 1.299(27)Å.[6] The benzimidazole moiety is planar with a maximum deviation of –0.017(2)Å for atom C4. The attached carbaldehyde and dienyl groups are also in planar with the best plane of the benzimidazole moiety. The sum of the angles (360.1°) at atom N1 is in accordance with sp^2 hybridization.

In addition to van der Waals forces, the molecular structure is stabilized by intramolecular C–H···O interactions. In the

Table 1 Crystal and experimental data

Chemical formula: $C_{11}H_8N_2O$
Formula weight = 184.19
T = 293(2)K
Crystal system: monoclinic Space group: $P2_1/n$
a = 3.9124(8)Å
b = 15.082(3)Å
c = 15.407(3)Å
↕= 91.784(4)°
V = 908.7(3)Å^3
Z = 4
D_x = 1.346 g/cm^3
Radiation: Mo K_α (λ = 0.71073 Å)
Crystal size = 0.24 × 0.22 × 0.20 mm^3
No. of reflections collected = 10174
θ range for data collection: 1.9 to 28.0°
Data/restraints/parameters = 2096/0/159
Goodness-of-fit on F^2 = 1.01
R indices [$I > 2\sigma(I)$]: $R1$ = 0.045, $wR2$ = 0.121
R indices (all data): $R1$ = 0.060, $wR2$ = 0.133
CCDC deposition number: 770251

Table 2 Atomic coordinates (Å × 10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) involving non-hydrogen atoms

Atom	*x*	*y*	*z*	$U_{(eq)}$
N(1)	1469(3)	867(1)	7707(1)	41(1)
C(1)	-119(3)	205(1)	7218(1)	41(1)
N(2)	-418(3)	-150(1)	8649(1)	53(1)
C(6)	-1254(3)	-420(1)	7815(1)	45(1)
C(7)	1190(4)	607(1)	8560(1)	47(1)
O(1)	4050(4)	1774(1)	9322(1)	79(1)
C(5)	-2957(4)	-1184(1)	7530(1)	54(1)
C(2)	-686(4)	97(1)	6329(1)	53(1)
C(3)	-2345(5)	-664(1)	6068(1)	61(1)
C(4)	-3442(4)	-1300(1)	6654(1)	59(1)
C(11)	2515(5)	1088(1)	9325(1)	63(1)
C(8)	3084(4)	1648(1)	7417(1)	47(1)
C(9)	3420(4)	1886(1)	6616(1)	50(1)
C(10)	3953(5)	2192(1)	5848(1)	68(1)

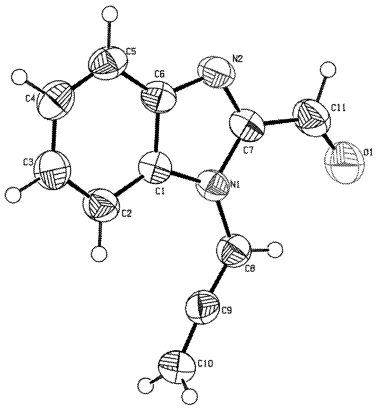

Fig. 2 ORTEP structure of the title compound, showing 50% probability ellipsoids.

Table 3 Hydrogen-bond geometry (Å, °)

D-H···A	D-H	H···A	D···A	D-H···A
C8-H8…O1	0.95(2)	2.25(2)	2.954(2)	130.2(1)
C10-H10A…O1^{i}	0.96(2)	2.46(2)	3.370(2)	157.7(2)
C11-H11…N2^{ii}	1.01(2)	2.55(2)	3.546(2)	169.5(2)

Symmetry codes: ^{i}*x*–1/2, –*y*+1/2, *z*–1/2; ^{ii}–*x*, –*y*, –*z*+2.

molecular packing, C–H···N hydrogen bonds link inversion-related molecules to form a $R_2^2(8)$ graph-set dimer. Atom H10A of C10 forms a intermolecular hydrogen bond with oxygen atom O1, forming a C–H···O hydrogen bond on either side of the dimer (Fig. 3). In addition to this, a weak $\pi\cdots\pi$ interaction between the imidazole ring (N1/C1/C6/N2/C7) at (*x*, *y*, *z*) and the benzene ring (C1–C6) at (1+*x*, *y*, *z*) stabilizes the molecular

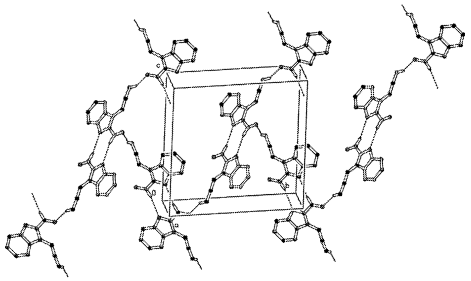

Fig. 3 Molecular packing viewed along the *a* axis; H-bonds are shown as dashed lines.

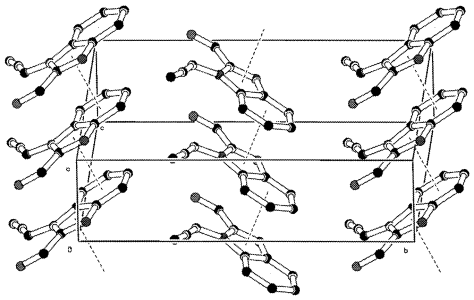

Fig. 4 Molecular packing showing $\pi\cdots\pi$ interactions.

packing (Fig. 4). The centroid-to-centroid distance is 3.762(1)Å. The geometrie of all hydrogen bonds are listed in Table 3.

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