Crystal structure of 1-(1*H*-benzimidazol-2-yl)-3-(3-nitrophenyl)prop-2-en-1-one, $C_{16}H_{11}N_3O_3$

Yvon Bibila Mayaya Bisseyou*[,I,II], Benoît Guillot[II], Drissa Sissouma[III], Claude Lecomte[II] and Jules Tenon Abodou[I]

[I] Université de Cocody, Laboratoire de Cristallographie et Physique Moléculaire, UFR SSMT, 22 BP 582 Abidjan 22, Côte d'Ivoire
[II] Nancy Université, Laboratoire de Cristallographie, Résonance Magnétique et Modélisations (CRM2, UMR CNRS 7036), Institut Jean Barriol, BP 70239, Boulevard des Aiguillettes, 54506 Vandoeuvre-lés Nancy, France
[III] Université de Cocody, Laboratoire de Chimie Organique Structurale, UFR SSMT, 22 BP 582 Abidjan 22, Côte d'Ivoire

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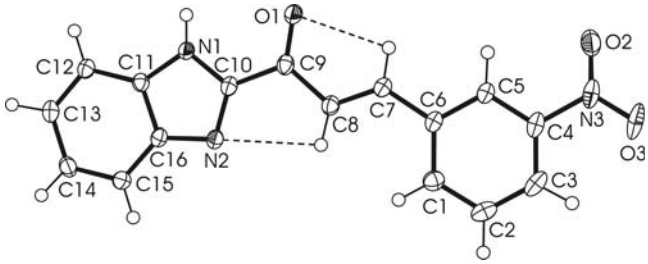

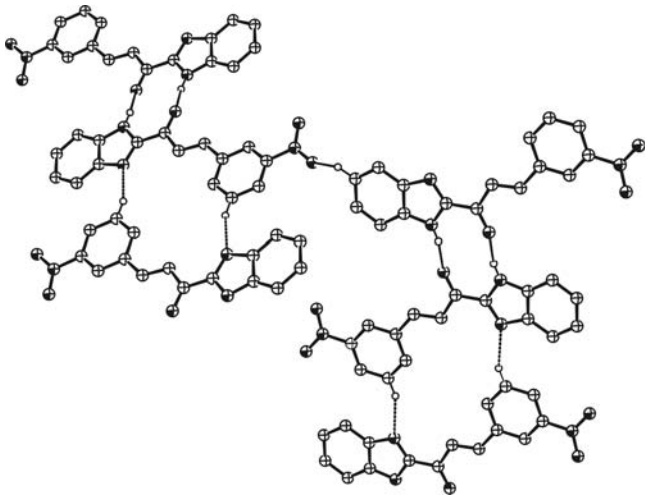

Abstract

$C_{16}H_{11}N_3O_3$, monoclinic, $P12_1/c1$ (no. 14), a = 9.3959(2) Å, b = 5.7085(1) Å, c = 24.8921(7) Å, β = 96.885(1)°, V = 1325.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.152, T = 100 K.

Source of material

2-Acetylbenzimidazole (1.5 g, 10 mmol) was dissolved to a solution of EtOH (40 ml) and 3 g (75 mmol) of NaOH. To this solution, 10 mmol of 3-nitrobenzaldehyde was added. The mixture was stirred at ambient temperature during 5 h. The resulting mixture was neutralized with a 4 N solution of HCl. The precipitate was dried and recrystallized from a mixture of toluene/EtOH (4 : 1, *v*/*v*) (yield 68 %, m.p. > 533 K). Yellow crystals of parallelepiped shape suitable for single-crystal diffraction were grown by slow evaporation at ambient temperature and in air from a hot solution in acetonitrile. NMR data are available in the CIF.

Experimental details

All H atoms were treated as riding atoms in geometrically idealized positions with d(C—H) = 0.95 - 0.97 Å, d(N—H) = 0.88 Å, and U_{iso}(H) = 1.2 - 1.4 U_{eq}(C,N).

Discussion

Benzimidazole and its derivatives constitute one of the most extensively studied families of organic compounds in regard to the number of articles published on these compounds. This great attention is essentially due to their wide range of biopharmacological activities including antihelminthic [1], antitumoral [2], antimicrobial [3], antifungal [4], antiviral [5], antihistaminic and antiallergic [6] properties. The benzimidazole derivatives showed to be also excellent inhibitors of HIV-1 reverse transcriptase [7]. The benzimidazole nucleus system, whose inestimable virtues seduced the scientific community, was moreover classified as an important pharmacophore in the discovery of modern drug [8]. From this point of view, the benzimidazole nucleus system constitutes a basic molecular scaffold in the design of new molecules of pharmacological interest.

The title molecular structure consists of three main parts: the benzimidazole ring and the nitrophenyl ring related by enone group (figure, top). In the benzimidazole ring the bond lengths and angles are in good agreement with those observed in the 1-(1*H*-benzimidazol-2-yl)-3-(3-chlorophenyl)-prop-2-en-1-one [9]. The C10=N2 double bond and C16—N2 single bond in the imidazole ring are clearly distinguishable with bond lengths of 1.323(2) and 1.387(1) Å, respectively. The benzimidazole ring makes dihedral angles of 2.66(3)° and 1.16(3)° with phenyl ring and mean plane of enone group, respectively. It confers to the molecule an almost planar structure. The magnitudes of structural parameters of phenyl ring are within normal ranges [10], with a light opening of intramolecular angle at C4 atom due to the presence of nitro group which is twisted of 13° with respect of phenyl ring. The N3—O2 and N3—O3 bonds of nitro group have similar distances of 1.226(2) and 1.233(1) Å in good agreement with those observed in electron density analysis of 1,3,4-trinitro-7,8-dizapentalene [11].

Intramolecular interactions analysis performed with PLATON [12] suggest existence of two intramolecular hydrogen bonds C7–H7···O1 (d(H7···O1) = 2.46 Å, d(C7···O1) = 2.7966 Å, ∠C7–H7···O1 = 101°) and C8–H8···N2 (d(H8···N2) = 2.60 Å,

* Correspondence author (e-mail: Yvon.Bibila@crm2.uhp-nancy.fr)

d(C8···N2)= 2.9437 Å, ∠C8–H8···N2 = 102°). Each of these hydrogen bonds generates an S(5) ring motif [13].

In the title crystal structure, molecules form a supramolecular network structure constituted by complex layers. Each layer is formed by combination of $R^2_2(10)$ and $R^2_2(18)$ basic motifs [14] arranged alternately and obtained from the following hydrogen bonds: N1–H9···O1^{i} (d(H9···O1) = 2.02 Å, d(N1···O1) = 2.824(2) Å and ∠N1–H9···O1 = 154°) and C2–H2···N2^{ii} (d(H2···N2) = 2.84 Å, d(C2···N2) = 3.702 Å and ∠C2–H2···N2 = 149°) (symmetry code i: 1–x,2–y,–z; ii: 2–x,2–y, 2–z), respectively. Within these layers, we note also the presence of H···H (d(H3···H15) = 2.266(4) Å) intermolecular interactions which are well known for their stabilizing nature. The layers forming stack in the block units and arranged in alternate fashion are stabilized by $\pi\cdots\pi$ (d(C2···C10) = 3.372(2) Å) and $\pi\cdots$C (d(C12···C7) = 3.293(2) Å) interactions. Each molecule in a layer is bound to next molecule of adjacent layer in [010] direction via an C14–H14···O3^{iii} intermolecular hydrogen bond: d(H14···O3) = 2.48 Å, d(C14···O3) = 3.144(2) Å, ∠C14–H14···O3 = 126°; symmetry code iii: 1+x,½–y,–½+z, so forming infinite chains (figure, bottom). Propagation of these chains by the space-group symmetry operations generates supramolecular structure controlled by hydrogen bonding.

Table 1. Data collection and handling.

Crystal:	yellow parallelepiped, size 0.28 × 0.49 × 0.70 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.05 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ
$2\theta_{max}$:	55.7°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	30696, 3155
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2905
$N(param)_{refined}$:	200
Programs:	PLATON [12], DENZO [14], CRYSTALS [15], SIR92 [16], WinGX [17], ORTEP [18]

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

Atom	Site	x	y	z	U_{iso}
H(5)	4*e*	0.9595	0.3649	0.8399	0.0242
H(7)	4*e*	0.8192	0.3046	0.9110	0.0243
H(8)	4*e*	0.8142	0.6251	0.9963	0.0249
H(13)	4*e*	0.3149	0.3665	1.2134	0.0271
H(1)	4*e*	0.9655	0.8676	0.9569	0.0285
H(12)	4*e*	0.3492	0.1106	1.1417	0.0250
H(9)	4*e*	0.5001	0.0964	1.0492	0.0273
H(15)	4*e*	0.5979	0.8376	1.1629	0.0303
H(14)	4*e*	0.4359	0.7221	1.2232	0.0310
H(3)	4*e*	1.1987	0.9490	0.8303	0.0339
H(2)	4*e*	1.1259	1.0887	0.9124	0.0364

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}
O(1)	4*e*	0.66190(9)	0.1299(2)	0.97184(4)	0.0286(5)	0.0355(6)	0.0215(5)	–0.0130(4)	0.0091(3)	–0.0070(4)
O(2)	4*e*	1.1148(1)	0.3472(2)	0.76883(4)	0.0296(5)	0.0487(7)	0.0273(5)	–0.0032(4)	0.0105(4)	–0.0047(4)
O(3)	4*e*	1.1977(1)	0.6904(2)	0.75211(5)	0.0352(6)	0.0590(7)	0.0420(6)	0.0018(5)	0.0244(5)	0.0239(5)
N(1)	4*e*	0.5362(1)	0.2243(2)	1.06480(4)	0.0214(5)	0.0238(6)	0.0161(5)	–0.0065(4)	0.0058(4)	–0.0029(4)
N(2)	4*e*	0.6536(1)	0.5678(2)	1.07415(4)	0.0184(5)	0.0214(5)	0.0200(5)	0.0001(4)	0.0059(4)	0.0015(4)
N(3)	4*e*	1.1358(1)	0.5544(2)	0.78004(4)	0.0189(5)	0.0431(7)	0.0242(5)	0.0011(4)	0.0082(4)	0.0107(5)
C(11)	4*e*	0.4957(1)	0.3253(2)	1.11059(4)	0.0174(5)	0.0211(6)	0.0155(5)	–0.0003(4)	0.0017(4)	–0.0001(4)
C(1)	4*e*	0.9962(1)	0.8103(2)	0.92387(5)	0.0257(6)	0.0215(6)	0.0243(6)	–0.0010(5)	0.0008(5)	0.0025(4)
C(12)	4*e*	0.4000(1)	0.2552(2)	1.14641(4)	0.0190(5)	0.0230(6)	0.0195(6)	–0.0016(4)	0.0038(4)	0.0026(5)
C(8)	4*e*	0.7924(1)	0.4877(2)	0.97562(5)	0.0199(5)	0.0248(6)	0.0182(6)	–0.0019(5)	0.0036(4)	0.0010(4)
C(10)	4*e*	0.6291(1)	0.3753(2)	1.04485(5)	0.0168(5)	0.0249(6)	0.0174(6)	–0.0019(4)	0.0028(4)	0.0030(4)
C(16)	4*e*	0.5699(1)	0.5403(2)	1.11598(5)	0.0153(5)	0.0210(6)	0.0196(5)	0.0001(4)	0.0035(4)	0.0023(4)
C(5)	4*e*	0.9920(1)	0.5107(2)	0.85530(5)	0.0159(5)	0.0246(6)	0.0183(5)	–0.0010(4)	0.0031(4)	0.0040(4)
C(3)	4*e*	1.1350(1)	0.8613(2)	0.84933(6)	0.0203(6)	0.0306(7)	0.0345(7)	–0.0045(5)	0.0016(5)	0.0145(5)
C(4)	4*e*	1.0858(1)	0.6469(2)	0.82967(5)	0.0170(5)	0.0311(7)	0.0214(6)	0.0007(5)	0.0048(4)	0.0097(5)
C(9)	4*e*	0.6931(1)	0.3164(2)	0.99526(5)	0.0181(5)	0.0267(7)	0.0168(5)	–0.0030(4)	0.0020(4)	0.0008(4)
C(15)	4*e*	0.5491(1)	0.6912(2)	1.15882(5)	0.0200(6)	0.0215(6)	0.0309(6)	–0.0020(4)	0.0072(5)	–0.0041(5)
C(7)	4*e*	0.8471(1)	0.4455(2)	0.92944(5)	0.0174(5)	0.0229(6)	0.0194(6)	–0.0020(4)	0.0025(4)	0.0027(4)
C(2)	4*e*	1.0908(1)	0.9419(2)	0.89733(6)	0.0288(7)	0.0217(7)	0.0344(7)	–0.0055(5)	–0.0015(5)	0.0060(5)
C(6)	4*e*	0.9458(1)	0.5926(2)	0.90311(5)	0.0170(5)	0.0215(6)	0.0181(5)	–0.0003(4)	0.0026(4)	0.0033(4)
C(14)	4*e*	0.4542(1)	0.6225(2)	1.19392(5)	0.0212(6)	0.0310(7)	0.0243(6)	0.0013(5)	0.0067(4)	–0.0065(5)
C(13)	4*e*	0.3810(1)	0.4062(2)	1.18792(5)	0.0181(5)	0.0293(7)	0.0192(5)	0.0019(4)	0.0042(4)	0.0025(5)

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