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Second monoclinic polymorph of 2-(benzimidazol-2-yl)-6-methoxyphenol

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The asymmetric unit of the title compound, $C_{14}H_{12}N_2O_2$, contains four crystallographically independent molecules. The crystal packing is stabilized by π–π, N—H···O and C—H···π interactions.

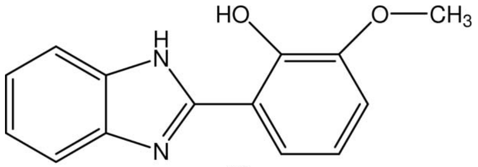
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.008 Å
R factor = 0.091
wR factor = 0.223
Data-to-parameter ratio = 12.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The benzimidazole skeleton possesses multiple pharmacological activities, such as antibacterial, antiviral, antitumour, antiplasmodium, anti-inflammatory, antiprotozoal, antihistamine and antifungal properties, and benzimidazole derivatives are used as drugs in both human and veterinary medicine (MacDonald *et al.*, 2004; Velík *et al.*, 2004; Vlahakis *et al.*, 2006). In this paper, we report the crystal structure of the title compound, (I). Although the crystal structure of (I) has been published previously (Elerman & Kabak, 1997) (space group $P2_1/n$, Z = 4), the present crystal structure is in a different space group ($P2/c$, Z = 16).



(I)

Bond lengths and angles in (I) display normal values (Allen *et al.*, 1987), comparable with those in the other polymorph (Elerman & Kabak, 1997). The dihedral angles between the benzene and benzimidazole ring systems in the four independent molecules, *i.e.* C8*A*–C13*A* and C1*A*–C6*A*/N1*A*/C7*A*/N2*A*, C8*B*–C13*B* and C1*B*–C6*B*/N1*B*/C7*B*/N2*B*, C8*C*–C13*C* and C1*C*–C6*C*/N1*C*/C7*C*/N2*C*, and C8*D*–C13*D* and C1*D*–C6*D*/N1*D*/C7*D*/N2*D* are 5.6 (2), 4.8 (2), 2.2 (3) and 4.8 (3)°, respectively. The methoxy groups are almost coplanar with the attached rings, with C14*A*—O2*A*—C12*A*—C11*A*, C14*B*—O2*B*—C12*B*—C11*B*, C14*C*—O2*C*—C12*C*—C11*C* and C14*D*—O2*D*—C12*D*—C11*D* torsion angles of −7.8 (8), −7.7 (8), −4.0 (9) and −0.1 (8)°, respectively. In each molecule, an intramolecular O—H···N interaction (Table 1) generates an S(6) ring (Bernstein *et al.*, 1995).

The crystal structure is stabilized by π–π interactions, in which the centroid–centroid distances between N1*A*/C7*A*/N2*A*/C1*A*/C6*A* at (x, y, z) and C8*A*–C13*A* at $(1 - x, y, \frac{1}{2} - z)$, N1*B*/C7*B*/N2*B*/C1*B*/C6*B* at (x, y, z) and C8*B*–C13*B* at $(-x, y, \frac{1}{2} - z)$, N1*C*/C7*C*/N2*C*/C1*C*/C6*C* at (x, y, z) and C8*C*–C13*C* at $(-x, y, \frac{1}{2} - z)$, N1*D*/C7*D*/N2*D*/C1*D*/C6*D* at (x, y, z) and C8*D*–C13*D* at $(1 - x, y, \frac{1}{2} - z)$, C1*B*–C6*B* at (x, y, z) and C8*B*–C13*B* at $(-x, y, \frac{1}{2} - z)$, and C1*D*–C6*D* at (x, y, z) and C8*D*–C13*D* at

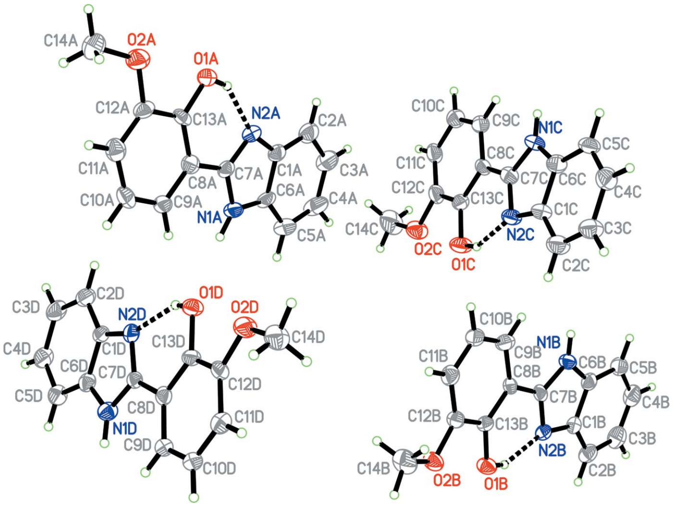


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The intramolecular hydrogen bonds are shown as dashed lines.

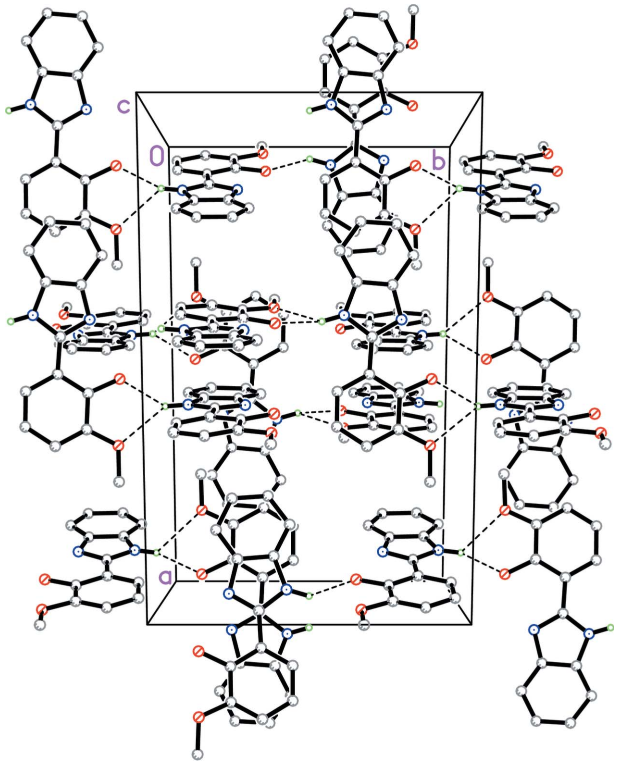


Figure 2
The crystal packing of (I), viewed down the c axis. The N—H···O hydrogen bonds are shown as dashed lines. H atoms not involved in the hydrogen bonds have been omitted for clarity.

$(1 - x, y, \frac{1}{2} - z)$ are 3.503 (3), 3.548 (3), 3.545 (4), 3.533 (4), 3.628 (3) and 3.621 (4) Å, respectively. The crystal structure is further stabilized by N—H···O and C—H···π interactions (Table 1), the latter involving the N1*D*/C7*D*/N2*D*/C1*D*/C6*D*, N1*C*/C7*C*/N2*C*/C1*C*/C6*C*, C1*D*–C6*D*, C8*A*–C13*A* and C1*B*–C6*B* rings whose centroids are *Cg*1, *Cg*2, *Cg*3, *Cg*4 and *Cg*5, respectively.

Experimental

To a solution of *o*-phenylenediamine (0.216 g, 2 mmol) in acetonitrile (30 ml), *o*-vanillin (0.616 g, 4 mmol) was added. The mixture was refluxed with stirring for half an hour. The resultant red solution was filtered and allowed to evaporate slowly at room temperature. Crystals suitable for X-ray diffraction were formed after several weeks (m.p. 553–555 K). IR spectroscopy (KBr, ν cm^{-1}): 3482 (O—H), 3275 (N—H), 3066, 2995, 2960, 2933 (C—H), 1625 (C═N), 1592, 1534, 1744 (C═C), 1252 (C—N).

Crystal data

$C_{14}H_{12}N_2O_2$
$M_r = 240.26$
Monoclinic, $P2/c$
$a = 20.0313$ (6) Å
$b = 12.0577$ (4) Å
$c = 20.0379$ (7) Å
$\beta = 107.382$ (2)°
$V = 4618.8$ (3) Å^3

$Z = 16$
$D_x = 1.382$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 100.0$ (1) K
Block, yellow
0.25 × 0.25 × 0.25 mm

Data collection

Brucker SMART APEX2 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min} = 0.964$, $T_{\max} = 0.977$

31231 measured reflections
8132 independent reflections
5426 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.060$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.091$
$wR(F^2) = 0.223$
$S = 1.16$
8132 reflections
653 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0176P)^2 + 23.9891P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1, *Cg*2, *Cg*3, *Cg*4 and *Cg*5 are the centroids of the N1*D*/C7*D*/N2*D*/C1*D*/C6*D*, N1*C*/C7*C*/N2*C*/C1*C*/C6*C*, C1*D*–C6*D*, C8*A*–C13*A* and C1*B*–C6*B* rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*AB*···N2*A*	0.82	1.84	2.577 (6)	148
N1*A*—H1*AA*···O1*D*$^{\rm i}$	0.86	2.06	2.821 (6)	147
N1*A*—H1*AA*···O2*D*$^{\rm i}$	0.86	2.56	3.220 (6)	135
O1*B*—H1*BA*···N2*B*	0.82	1.81	2.546 (6)	149
N1*B*—H1*BB*···O1*C*$^{\rm ii}$	0.86	2.10	2.813 (6)	140
O1*C*—H1*CA*···N2*C*	0.82	1.85	2.592 (6)	149
N1*C*—H1*CB*···O1*B*$^{\rm iii}$	0.86	2.08	2.844 (6)	148
N1*C*—H1*CB*···O2*B*$^{\rm iii}$	0.86	2.50	3.108 (6)	128
O1*D*—H1*DA*···N2*D*	0.82	1.81	2.547 (5)	148
N1*D*—H1*DB*···O1*A*$^{\rm iv}$	0.86	2.12	2.832 (6)	140
N1*D*—H1*DB*···O2*A*$^{\rm iv}$	0.86	2.45	3.149 (6)	139
C3*B*—H3*BA*···*Cg*1$^{\rm v}$	0.93	3.03	3.815 (7)	143
C14*A*—H14*C*···*Cg*2$^{\rm i}$	0.96	3.07	3.777 (7)	131
C14*B*—H14*E*···*Cg*3$^{\rm i}$	0.96	2.93	3.417 (7)	112
C14*D*—H14*K*···*Cg*4$^{\rm vi}$	0.96	3.04	3.775 (6)	134
C3*C*—H3*CA*···*Cg*5$^{\rm vii}$	0.93	2.80	3.562 (6)	140

Symmetry codes: (i) $-x + 1, y, -z + \frac{1}{2}$; (ii) $-x, y, -z + \frac{1}{2}$; (iii) $x, y - 1, z$; (iv) $x, y + 1, z$; (v) $x - 1, y, z$; (vi) $x, -y + 1, z + \frac{1}{2}$; (vii) $-x, -y + 1, -z + 1$.

H atoms were positioned geometrically (O—H = 0.82 Å, N—H = 0.86 Å and C—H = 0.93–0.96 Å) and treated as riding, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(N,C) or $1.5U_{\rm eq}$(O and methyl C).

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Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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