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Key indicators

Single-crystal X-ray study
T = 100 K
Mean $\sigma(\text{C}-\text{C}) = 0.001 \text{ \AA}$
R factor = 0.050
wR factor = 0.137
Data-to-parameter ratio = 34.6

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

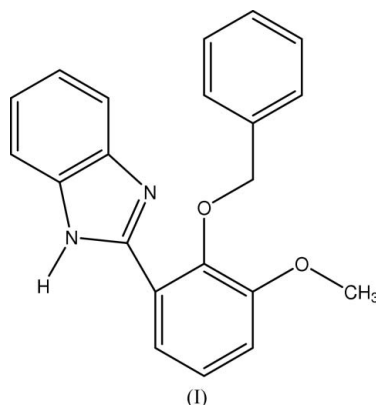
2-(2-Benzyloxy-3-methoxyphenyl)-1H-benzimidazole

In the title molecule, $\text{C}_{21}\text{H}_{18}\text{N}_2\text{O}_2$, all bond lengths and angles are normal. Weak intermolecular $\text{N}-\text{H} \cdots \text{N}$ hydrogen bonds link the molecules into chains along the *c* axis. The crystal packing is further stabilized by van der Waals forces.

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Comment

Benzimidazole and its derivatives are widely used in biological systems (Craigo *et al.*, 1999; Gudmundsson *et al.*, 2000; Trivedi *et al.*, 2006). They are often used in an experimental synthetic search for new drugs (Townsend & Revankav, 1970; Trivedi *et al.*, 2006). Some derivatives of benzimidazole are used as topoisomerase I inhibitors (Kim *et al.*, 1996), and as antitumor (Craigo *et al.*, 1999), antiviral (Gudmundsson *et al.*, 2000) and antibacterial (Khalafi-Nezhad *et al.*, 2005) agents. The title compound, (I), is a new benzimidazole derivative. We present here its crystal structure.



Bond lengths and angles in (I) show normal values (Allen *et al.*, 1987) and are comparable with those reported for the related structures (Beauchamp *et al.*, 1987). The methoxy group at C12 is almost coplanar with the attached ring [$\text{C11}-\text{C12}-\text{O2}-\text{C21} = -3.25(11)^\circ$], while the benzyloxy substituent is twisted away from the attached ring, with a $\text{C13}-\text{O1}-\text{C14}-\text{C15}$ torsion angle of $-135.66(7)^\circ$. Intramolecular $\text{N1}-\text{H1A} \cdots \text{O1}$ hydrogen bonds (Fig. 1 and Table 1) generate *S*(6) ring motifs (Bernstein *et al.*, 1995).

Weak intermolecular $\text{N}-\text{H} \cdots \text{N}$ hydrogen bonds (Table 1) link the molecules into chains extending along the *c* axis. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

Experimental

A 100 ml three-necked round-bottomed flask was equipped with a nitrogen inlet adapter, rubber septum, glass stopper and magnetic

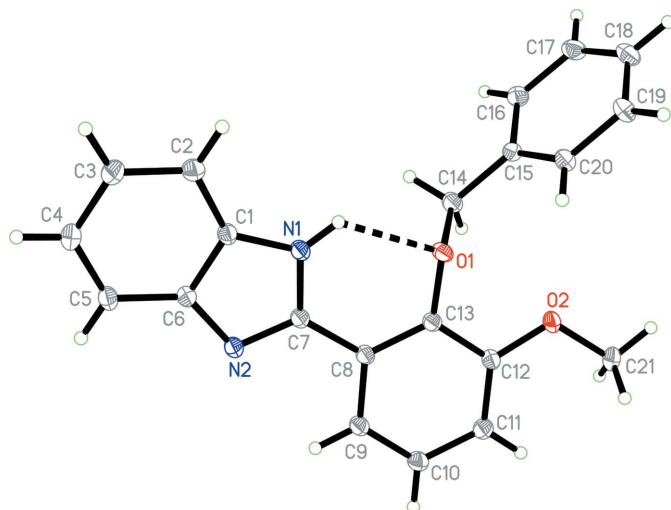


Figure 1
View of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed line indicates an intramolecular hydrogen bond.

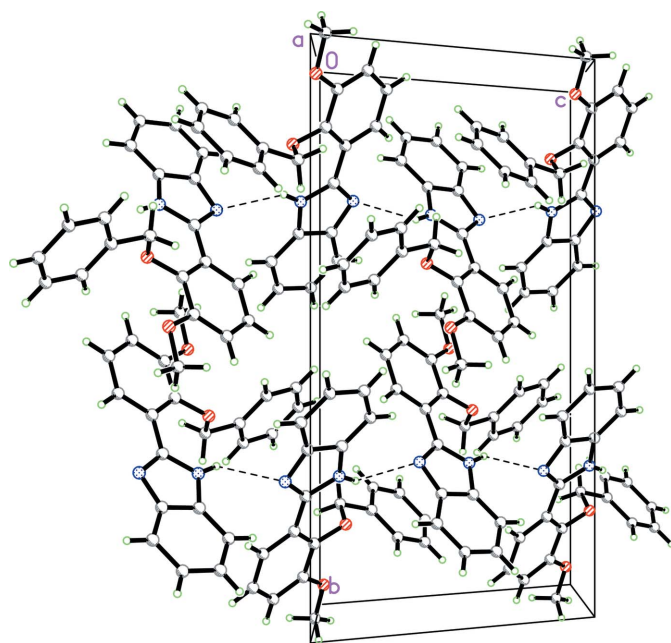


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

stirring bar. The flask was charged with 5 ml of dichloromethane and benzyl-*o*-vanillin (484.6 mg, 2 mmol) and was cooled in an ice–water bath while a solution of *o*-phenylenediamine (216.3 mg, 2 mmol) in 5 ml dichloromethane was added dropwise *via* a syringe over 15 min. After 30 min, 10 mg anhydrous magnesium sulfate was added in one portion. The ice–water bath was removed, and the reaction mixture was stirred at room temperature for 2 h. The resulting mixture was then filtered through a sintered glass funnel with the aid of two 10 ml portions of dichloromethane; the filtrate was concentrated at reduced pressure by rotary evaporation at room temperature, affording a yellowish brown syrup. This material was dissolved in 150 ml of ethanol heated in an 353 K water bath while 270 ml of hot water was

added with stirring. The resulting solution was allowed to cool to room temperature and was then cooled in an ice–water bath for 2 h. Filtration provided a light yellow powder of (I). The product was then purified by column chromatography with 30% ethanol in diethyl ether. Single crystals suitable for X-ray diffraction were obtained from ethanol–acetone (99:1 *v/v*).

Crystal data

$C_{21}H_{18}N_2O_2$
 $M_r = 330.37$
Monoclinic, $P2_1/c$
 $a = 9.5417$ (1) Å
 $b = 18.4590$ (3) Å
 $c = 11.0653$ (2) Å
 $\beta = 123.814$ (1)°
 $V = 1619.27$ (4) Å³

$Z = 4$
 $D_x = 1.355$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 100.0$ (1) K
Block, yellow
 $0.61 \times 0.28 \times 0.22$ mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.893$, $T_{\max} = 0.981$

54869 measured reflections
8446 independent reflections
6691 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.054$
 $\theta_{\text{max}} = 37.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.137$
 $S = 1.06$
8446 reflections
244 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 0.2664P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.61$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1A···O1	0.86	2.14	2.693 (1)	122
N1–H1A···N2 ⁱ	0.86	2.57	3.313 (1)	145

Symmetry code: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$.

H atoms were placed in calculated positions, with C–H = 0.93–0.97 Å and N–H = 0.86 Å. The H atoms were refined as riding and the U_{iso} values were freely refined.

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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