

2-Methoxy-6-(6-methyl-1*H*-benzimidazol-2-yl)phenol

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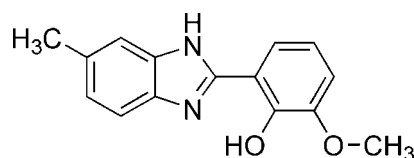
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.056; wR factor = 0.169; data-to-parameter ratio = 14.4.

The molecule of the title compound, $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_2$ is almost planar, the dihedral angle between the 6-methyl-1*H*-benzimidazole plane and the 2-methoxyphenol plane being $6.9(2)^\circ$. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond is present. Adjacent molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into a three-dimensional network structure. The benzoimidazole methyl group and its attached C atom are positionally disordered in a $0.724(4):0.276(4)$ ratio.

Related literature

For background to imidazole and its derivatives, see: Huang *et al.* (2004) and to benzimidazoles, see: Perry & Wilson (1993). For related structures, see: Savall & Fontimayor (2008).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_2$	$V = 2676.1(7) \text{ \AA}^3$
$M_r = 254.28$	$Z = 8$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 17.986(3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 11.4452(16) \text{ \AA}$	$T = 293 \text{ K}$
$c = 13.4105(19) \text{ \AA}$	$0.21 \times 0.17 \times 0.13 \text{ mm}$
$\beta = 104.216(2)^\circ$	

Data collection

Bruker APEXII CCD area-detector diffractometer	6836 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	2531 independent reflections
$T_{\min} = 0.982$, $T_{\max} = 0.989$	1441 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$	3 restraints
$wR(F^2) = 0.169$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
2531 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
176 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N2}$	0.82	1.83	2.567(2)	148
$\text{N1}-\text{H1A}\cdots\text{O2}^i$	0.92	2.54	3.173(3)	127
$\text{N1}-\text{H1A}\cdots\text{O1}^i$	0.92	2.06	2.920(3)	155

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2503).

References

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supplementary materials

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2-Methoxy-6-(6-methyl-1*H*-benzimidazol-2-yl)phenol

H.-Q. Xiao, M.-Z. Zhang and W. Wang

Comment

Imidazole and its derivatives are an important class of heterocycle with N-donor atoms, therefore they can be excellent organic ligands to generate various complexes (Huang *et al.*, 2004). Benzimidazoles are privileged structural units not only in the pharmaceutical industry but also in several other fields such as agricultural, electronic, and polymer chemistry (Perry *et al.*, 1993). We report here the synthesis and crystal structure of the title compound.

The molecular structure is shown in Fig. 1. The values of the geometric parameters in the compound are normal (Savall *et al.*, 2008) (Table 1). The benzimidazole and phenol groups are nearly coplanar, the dihedral angle between 6-methyl-1*H*-benzimidazole plane and 2-methoxyphenol plane is 6.9 (2)°. The compounds are linked by N—H···O hydrogen bonds [N1—H1A···O1, N1—H1A···O2, O1—H1···N2] into a three-dimensional network structure.

Experimental

A mixture of 4-methylbenzene-1,2-diamine (1 mmol) and 2-hydroxy-3-methoxybenzaldehyde (1 mmol) in ethanol (15 ml) was stirred for 8 h and then filtered. The resulting clear orange solution was vapor at room temperature for 7 d, after which orange block-shaped crystals of the title complex suitable for X-ray diffraction analysis were obtained, yield 45%.

Refinement

The H atoms were fixed geometrically and were treated as riding on their parent C atoms, with C—H distances in the range of 0.93–0.96 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$, or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The coordinates of the H atoms of the N—H and O—H groups were found from difference Fourier maps and were allowed for as riding atoms with O—H 0.82 Å and N—H 0.92 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$.

Figures

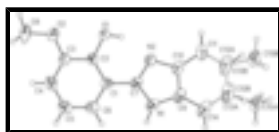


Fig. 1. The independent molecules of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

2-Methoxy-6-(6-methyl-1*H*-benzimidazol-2-yl)phenol

Crystal data

$\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_2$

$M_r = 254.28$

Monoclinic, $C2/c$

$F_{000} = 1072$

$D_x = 1.262 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

supplementary materials

Hall symbol: -C 2yc	$\lambda = 0.71073 \text{ \AA}$
$a = 17.986 (3) \text{ \AA}$	Cell parameters from 2318 reflections
$b = 11.4452 (16) \text{ \AA}$	$\theta = 2.4\text{--}23.9^\circ$
$c = 13.4105 (19) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 104.216 (2)^\circ$	$T = 293 \text{ K}$
$V = 2676.1 (7) \text{ \AA}^3$	Block, yellow
$Z = 8$	$0.21 \times 0.17 \times 0.13 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector diffractometer	2531 independent reflections
Radiation source: fine-focus sealed tube	1441 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.031$
$T = 293 \text{ K}$	$\theta_{\text{max}} = 25.7^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -21 \rightarrow 11$
$T_{\text{min}} = 0.982, T_{\text{max}} = 0.989$	$k = -13 \rightarrow 13$
6836 measured reflections	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.056$	H-atom parameters constrained
$wR(F^2) = 0.169$	$w = 1/[\sigma^2(F_o^2) + (0.0769P)^2 + 0.8329P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2531 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
176 parameters	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
3 restraints	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.34998 (10)	0.96117 (16)	0.14576 (12)	0.0718 (6)	
H1	0.3809	0.9222	0.1879	0.086*	
O2	0.23740 (12)	1.0753 (2)	0.03072 (14)	0.0942 (7)	
N1	0.39603 (12)	0.94335 (19)	0.46813 (14)	0.0644 (6)	
H1A	0.3689	0.9806	0.5087	0.077*	
N2	0.42667 (11)	0.89053 (17)	0.32288 (14)	0.0568 (5)	
C1	0.31924 (14)	1.0273 (2)	0.30233 (18)	0.0568 (6)	
C2	0.30615 (14)	1.0242 (2)	0.19534 (18)	0.0574 (6)	
C3	0.24529 (16)	1.0877 (2)	0.1342 (2)	0.0691 (8)	
C4	0.20037 (17)	1.1559 (3)	0.1795 (2)	0.0839 (9)	
H4	0.1605	1.1992	0.1389	0.101*	
C5	0.21404 (19)	1.1607 (3)	0.2856 (3)	0.0928 (10)	
H5	0.1834	1.2075	0.3158	0.111*	
C6	0.27225 (17)	1.0974 (3)	0.3463 (2)	0.0797 (9)	
H6	0.2807	1.1009	0.4175	0.096*	
C7	0.38041 (14)	0.9559 (2)	0.36368 (17)	0.0554 (6)	
C8	0.1738 (2)	1.1332 (4)	-0.0374 (3)	0.1217 (14)	
H8A	0.1748	1.1176	-0.1074	0.183*	
H8B	0.1776	1.2158	-0.0251	0.183*	
H8C	0.1266	1.1047	-0.0252	0.183*	
C9	0.45596 (15)	0.8650 (2)	0.49645 (19)	0.0659 (7)	
C10	0.47445 (14)	0.8322 (2)	0.40546 (19)	0.0618 (7)	
C11	0.53215 (16)	0.7501 (3)	0.4079 (2)	0.0806 (9)	
H11	0.5454	0.7270	0.3480	0.097*	
C14	0.49349 (18)	0.8175 (3)	0.5915 (2)	0.0865 (10)	
H14	0.4807	0.8398	0.6519	0.104*	
C12A	0.56835 (19)	0.7052 (3)	0.5008 (3)	0.1024 (16)	0.724 (4)
H12A	0.6072	0.6509	0.5033	0.123*	0.724 (4)
C13A	0.55061 (19)	0.7359 (3)	0.5920 (3)	0.0988 (17)	0.724 (4)
C15A	0.5957 (3)	0.6706 (5)	0.6876 (3)	0.1041 (15)	0.724 (4)
H15A	0.5797	0.6974	0.7469	0.156*	0.724 (4)
H15B	0.6495	0.6854	0.6969	0.156*	0.724 (4)
H15C	0.5861	0.5883	0.6788	0.156*	0.724 (4)
C13B	0.56835 (19)	0.7052 (3)	0.5008 (3)	0.0988 (17)	0.276 (4)
C12B	0.55061 (19)	0.7359 (3)	0.5920 (3)	0.1024 (16)	0.276 (4)
H12B	0.5769	0.7018	0.6536	0.123*	0.276 (4)
C15B	0.6306 (6)	0.6287 (11)	0.5397 (9)	0.1041 (15)	0.276 (4)
H15G	0.6498	0.5992	0.4838	0.156*	0.276 (4)
H15D	0.6136	0.5648	0.5748	0.156*	0.276 (4)
H15E	0.6707	0.6703	0.5868	0.156*	0.276 (4)

Atomic displacement parameters (\AA^2)

U^{11} U^{22} U^{33} U^{12} U^{13} U^{23}

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O1	0.0768 (12)	0.0922 (13)	0.0482 (10)	0.0351 (10)	0.0186 (9)	0.0116 (9)
O2	0.0935 (15)	0.1259 (18)	0.0566 (12)	0.0478 (13)	0.0059 (10)	0.0170 (11)
N1	0.0690 (14)	0.0807 (15)	0.0447 (12)	-0.0114 (12)	0.0164 (10)	0.0033 (11)
N2	0.0514 (12)	0.0654 (13)	0.0528 (12)	0.0031 (10)	0.0112 (10)	0.0091 (10)
C1	0.0562 (15)	0.0652 (15)	0.0518 (14)	0.0051 (13)	0.0186 (12)	0.0053 (12)
C2	0.0553 (14)	0.0654 (15)	0.0550 (15)	0.0130 (13)	0.0205 (12)	0.0091 (12)
C3	0.0693 (17)	0.0807 (19)	0.0588 (16)	0.0191 (15)	0.0185 (14)	0.0146 (14)
C4	0.0738 (19)	0.094 (2)	0.088 (2)	0.0316 (17)	0.0277 (17)	0.0206 (17)
C5	0.097 (2)	0.103 (2)	0.091 (2)	0.038 (2)	0.046 (2)	0.0105 (19)
C6	0.090 (2)	0.094 (2)	0.0645 (17)	0.0202 (18)	0.0352 (16)	0.0058 (15)
C7	0.0560 (14)	0.0641 (15)	0.0473 (14)	-0.0055 (13)	0.0147 (12)	0.0061 (11)
C8	0.116 (3)	0.151 (3)	0.080 (2)	0.059 (3)	-0.010 (2)	0.031 (2)
C9	0.0529 (15)	0.0782 (18)	0.0590 (16)	-0.0160 (14)	-0.0009 (13)	0.0180 (13)
C10	0.0528 (15)	0.0712 (17)	0.0565 (16)	-0.0081 (13)	0.0042 (12)	0.0166 (13)
C11	0.0611 (17)	0.086 (2)	0.087 (2)	0.0102 (16)	0.0036 (15)	0.0235 (16)
C14	0.079 (2)	0.112 (2)	0.0576 (17)	-0.0321 (19)	-0.0033 (15)	0.0269 (16)
C12A	0.080 (4)	0.103 (4)	0.116 (3)	0.015 (3)	0.006 (3)	0.042 (3)
C13A	0.070 (3)	0.129 (5)	0.077 (2)	-0.016 (4)	-0.020 (2)	0.055 (3)
C15A	0.101 (3)	0.131 (4)	0.072 (3)	0.028 (3)	0.005 (2)	0.034 (2)
C13B	0.070 (3)	0.129 (5)	0.077 (2)	-0.016 (4)	-0.020 (2)	0.055 (3)
C12B	0.080 (4)	0.103 (4)	0.116 (3)	0.015 (3)	0.006 (3)	0.042 (3)
C15B	0.101 (3)	0.131 (4)	0.072 (3)	0.028 (3)	0.005 (2)	0.034 (2)

Geometric parameters (Å, °)

O1—C2	1.357 (3)	C8—H8A	0.9600
O1—H1	0.8200	C8—H8B	0.9600
O2—C3	1.367 (3)	C8—H8C	0.9600
O2—C8	1.438 (3)	C9—C10	1.393 (4)
N1—C7	1.367 (3)	C9—C14	1.397 (3)
N1—C9	1.382 (3)	C10—C11	1.394 (4)
N1—H1A	0.9194	C11—C12A	1.357 (4)
N2—C7	1.332 (3)	C11—H11	0.9300
N2—C10	1.394 (3)	C14—C13A	1.388 (5)
C1—C2	1.396 (3)	C14—H14	0.9300
C1—C6	1.397 (4)	C12A—C13A	1.383 (5)
C1—C7	1.453 (3)	C12A—H12A	0.9300
C2—C3	1.399 (3)	C13A—C15A	1.531 (4)
C3—C4	1.368 (4)	C15A—H15A	0.9600
C4—C5	1.384 (4)	C15A—H15B	0.9600
C4—H4	0.9300	C15A—H15C	0.9600
C5—C6	1.366 (4)	C15B—H15G	0.9600
C5—H5	0.9300	C15B—H15D	0.9600
C6—H6	0.9300	C15B—H15E	0.9600
C2—O1—H1	109.2	O2—C8—H8C	109.5
C3—O2—C8	117.7 (2)	H8A—C8—H8C	109.5
C7—N1—C9	107.4 (2)	H8B—C8—H8C	109.5
C7—N1—H1A	123.5	N1—C9—C10	105.8 (2)
C9—N1—H1A	129.0	N1—C9—C14	132.3 (3)

C7—N2—C10	105.6 (2)	C10—C9—C14	121.9 (3)
C2—C1—C6	118.8 (2)	C9—C10—C11	119.8 (2)
C2—C1—C7	118.8 (2)	C9—C10—N2	109.5 (2)
C6—C1—C7	122.5 (2)	C11—C10—N2	130.7 (3)
O1—C2—C1	123.0 (2)	C12A—C11—C10	117.6 (3)
O1—C2—C3	117.0 (2)	C12A—C11—H11	121.2
C1—C2—C3	120.0 (2)	C10—C11—H11	121.2
O2—C3—C4	125.8 (2)	C13A—C14—C9	117.3 (3)
O2—C3—C2	114.3 (2)	C13A—C14—H14	121.4
C4—C3—C2	119.9 (2)	C9—C14—H14	121.4
C3—C4—C5	120.3 (3)	C11—C12A—C13A	123.6 (3)
C3—C4—H4	119.9	C11—C12A—H12A	118.2
C5—C4—H4	119.9	C13A—C12A—H12A	118.2
C6—C5—C4	120.5 (3)	C12A—C13A—C14	119.8 (3)
C6—C5—H5	119.7	C12A—C13A—C15A	115.3 (4)
C4—C5—H5	119.7	C14—C13A—C15A	124.9 (4)
C5—C6—C1	120.6 (3)	C13A—C15A—H15A	109.5
C5—C6—H6	119.7	C13A—C15A—H15B	109.5
C1—C6—H6	119.7	H15A—C15A—H15B	109.5
N2—C7—N1	111.7 (2)	C13A—C15A—H15C	109.5
N2—C7—C1	123.1 (2)	H15A—C15A—H15C	109.5
N1—C7—C1	125.2 (2)	H15B—C15A—H15C	109.5
O2—C8—H8A	109.5	H15G—C15B—H15D	109.5
O2—C8—H8B	109.5	H15G—C15B—H15E	109.5
H8A—C8—H8B	109.5	H15D—C15B—H15E	109.5

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>
O1—H1...N2	0.82	1.83	2.567 (2)	148
N1—H1A...O2 ⁱ	0.92	2.54	3.173 (3)	127
N1—H1A...O1 ⁱ	0.92	2.06	2.920 (3)	155

Symmetry codes: (i) *x*, $-y+2$, $z+1/2$.

Fig. 1

