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3-Methoxy-4-[3-(2-methyl-4-nitro-1*H*-imidazol-1-yl)propoxy]benzaldehydeLei Jin,^{a,b} Guang-Zhou Wang^a and Cheng-He Zhou^{a*}

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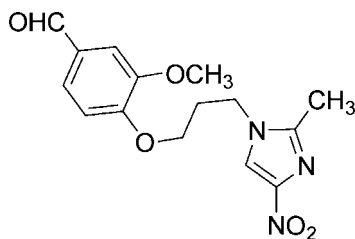
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.130; data-to-parameter ratio = 15.9.

In the title molecule, $\text{C}_{15}\text{H}_{17}\text{N}_3\text{O}_5$, the dihedral angle between the benzene and imidazole rings is $3.69(2)^\circ$. The crystal structure is stabilized by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and $\pi-\pi$ stacking interactions with a centroid-centroid distance of $3.614(1)$ Å.

Related literature

For general background to the biological activity of nitroimidazole and its derivatives, see: Demirayak *et al.* (1999); Huang *et al.* (2007); Olender *et al.* (2009). For the synthetic procedure, see: Khalafi-Nezhad *et al.* (2005).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{17}\text{N}_3\text{O}_5$
 $M_r = 319.32$
 Monoclinic, $P2_1/n$
 $a = 9.4885(14)$ Å
 $b = 13.048(2)$ Å

$c = 12.745(2)$ Å
 $\beta = 101.120(3)^\circ$
 $V = 1548.3(4)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.10$ mm⁻¹
 $T = 173$ K

0.28 × 0.24 × 0.2 mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.971$, $T_{\max} = 0.979$

7761 measured reflections
 3329 independent reflections
 2329 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.130$
 $S = 1.03$
 3329 reflections

210 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C4}-\text{H4B}\cdots\text{O4}^{\text{i}}$	0.98	2.58	3.415 (3)	144
$\text{C8}-\text{H8}\cdots\text{O4}^{\text{ii}}$	0.95	2.51	3.229 (2)	133
$\text{C10}-\text{H10B}\cdots\text{O2}^{\text{iii}}$	0.99	2.56	3.312 (2)	133
$\text{C10}-\text{H10A}\cdots\text{O1}^{\text{iv}}$	0.99	2.58	3.461 (2)	148
$\text{C14}-\text{H14}\cdots\text{O1}^{\text{iv}}$	0.95	2.29	3.166 (2)	153

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $x, y + 1, z$; (iii) $-x, -y + 1, -z$; (iv) $x - \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 1999); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2876).

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supplementary materials

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3-Methoxy-4-[3-(2-methyl-4-nitro-1*H*-imidazol-1-yl)propoxy]benzaldehyde

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Comment

Nitroimidazole and its derivatives possess several biological activities such as radiosensitizer, anti-tuberculosis and antimicrobial (Demirayak *et al.*, 1999; Huang *et al.*, 2007; Olender *et al.*, 2009). In view of the therapeutic potentials of nitroimidazole derivatives, we are interested in the research and development of nitroimidazole compounds as drugs. Herein we report the crystal structure of the title compound (I).

The structure of the title compound (I) is shown in Fig 1. In the molecule the dihedral angle between the benzene and imidazole rings is 3.69 (2)°. The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds and significant π – π stacking interactions with a centroid to centroid distance of 3.614 (1)Å between benzene and imidazole rings related by the symmetry operator (1/2-x, 1/2+y, 1/2-z).

Experimental

Compound (I) was synthesized according to the procedure of Khalafi-Nezhad *et al.* (2005). Single crystals used in X-ray diffraction studies were grown by slow evaporation at room temperature of solutions of (I) in ethyl acetate and dichloromethane mixtures.

Refinement

Hydrogen atoms were placed in calculated positions with C—H = 0.95Å (aromatic), 0.99Å (methylene) and 0.98Å (methyl) with $U_{iso}(H) = 1.2U_{eq}(C)$ (aromatic and methylene C) or $1.5U_{eq}(C)$ (methyl C).

Figures

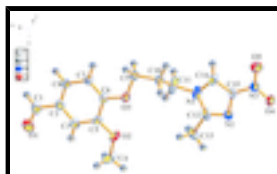


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

3-Methoxy-4-[3-(2-methyl-4-nitro-1*H*-imidazol-1-yl)propoxy]benzaldehyde

Crystal data

C₁₅H₁₇N₃O₅

$M_r = 319.32$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$F_{000} = 672$

$D_x = 1.370 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3149 reflections

supplementary materials

$a = 9.4885 (14) \text{ \AA}$	$\theta = 2.3\text{--}26.9^\circ$
$b = 13.048 (2) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 12.745 (2) \text{ \AA}$	$T = 173 \text{ K}$
$\beta = 101.120 (3)^\circ$	Block, colorless
$V = 1548.3 (4) \text{ \AA}^3$	$0.28 \times 0.24 \times 0.2 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	3329 independent reflections
Radiation source: fine-focus sealed tube	2329 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.022$
$T = 173 \text{ K}$	$\theta_{\text{max}} = 27.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 12$
$T_{\text{min}} = 0.971$, $T_{\text{max}} = 0.979$	$k = -15 \rightarrow 16$
7761 measured reflections	$l = -13 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.130$	$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 0.3992P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
3329 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
210 parameters	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
	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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2645 (2)	1.03088 (14)	-0.03024 (16)	0.0502 (5)
H1	0.2344	1.0901	0.0028	0.060*
C2	0.21471 (17)	0.93177 (12)	0.00141 (13)	0.0363 (4)
C3	0.25719 (18)	0.84045 (12)	-0.04102 (14)	0.0381 (4)
H3	0.3193	0.8421	-0.0912	0.046*
C4	0.3482 (2)	0.65169 (17)	-0.1102 (2)	0.0691 (7)
H4A	0.3107	0.6882	-0.1769	0.104*
H4B	0.3681	0.5803	-0.1262	0.104*
H4C	0.4370	0.6847	-0.0737	0.104*
C5	0.20870 (17)	0.74857 (12)	-0.00975 (13)	0.0346 (4)
C6	0.11391 (16)	0.74686 (11)	0.06315 (12)	0.0305 (3)
C7	0.07388 (17)	0.83703 (11)	0.10519 (13)	0.0322 (4)
H7	0.0114	0.8359	0.1551	0.039*
C8	0.12529 (17)	0.92983 (12)	0.07433 (13)	0.0347 (4)
H8	0.0985	0.9921	0.1038	0.042*
C9	-0.01627 (18)	0.64126 (12)	0.16566 (14)	0.0360 (4)
H9A	-0.1090	0.6775	0.1440	0.043*
H9B	0.0344	0.6699	0.2346	0.043*
C10	-0.04042 (17)	0.52728 (12)	0.17563 (14)	0.0378 (4)
H10A	-0.1107	0.5152	0.2225	0.045*
H10B	-0.0800	0.4981	0.1043	0.045*
C11	0.0990 (2)	0.47573 (13)	0.2218 (2)	0.0616 (6)
H11A	0.1721	0.4966	0.1802	0.074*
H11B	0.1316	0.4999	0.2962	0.074*
C12	0.16148 (17)	0.29675 (13)	0.16772 (13)	0.0384 (4)
C13	0.2589 (2)	0.33135 (16)	0.09670 (16)	0.0560 (5)
H13A	0.2936	0.2717	0.0624	0.084*
H13B	0.3407	0.3680	0.1390	0.084*
H13C	0.2066	0.3772	0.0417	0.084*
C14	0.01304 (17)	0.30668 (12)	0.28026 (15)	0.0403 (4)
H14	-0.0475	0.3304	0.3264	0.048*
C15	0.04200 (16)	0.20834 (11)	0.25747 (13)	0.0326 (4)
N1	0.09014 (14)	0.36345 (10)	0.22222 (12)	0.0417 (4)
N2	0.13429 (14)	0.20076 (10)	0.18821 (11)	0.0346 (3)
N3	-0.01634 (15)	0.11998 (11)	0.29957 (12)	0.0404 (4)
O1	0.34061 (19)	1.04390 (12)	-0.09476 (13)	0.0758 (5)
O2	0.24432 (13)	0.65445 (9)	-0.04271 (11)	0.0493 (4)
O3	0.06945 (12)	0.65190 (8)	0.08547 (10)	0.0376 (3)
O4	0.02197 (15)	0.03485 (9)	0.27613 (12)	0.0544 (4)
O5	-0.10418 (15)	0.13411 (11)	0.35800 (12)	0.0611 (4)

Atomic displacement parameters (\AA^2)

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

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C1	0.0631 (12)	0.0379 (10)	0.0515 (11)	-0.0115 (8)	0.0156 (10)	0.0021 (8)
C2	0.0422 (9)	0.0306 (8)	0.0360 (9)	-0.0055 (7)	0.0074 (7)	0.0004 (7)
C3	0.0385 (9)	0.0378 (9)	0.0404 (9)	-0.0086 (7)	0.0139 (7)	-0.0058 (7)
C4	0.0556 (12)	0.0577 (13)	0.1071 (19)	-0.0074 (10)	0.0486 (13)	-0.0360 (13)
C5	0.0327 (8)	0.0294 (8)	0.0427 (9)	-0.0027 (6)	0.0097 (7)	-0.0106 (7)
C6	0.0306 (8)	0.0248 (7)	0.0355 (8)	-0.0027 (6)	0.0046 (6)	-0.0010 (6)
C7	0.0370 (8)	0.0285 (8)	0.0327 (8)	0.0011 (6)	0.0106 (7)	-0.0008 (6)
C8	0.0420 (9)	0.0249 (8)	0.0365 (9)	0.0012 (6)	0.0057 (7)	-0.0022 (6)
C9	0.0391 (9)	0.0279 (8)	0.0435 (9)	-0.0007 (7)	0.0140 (7)	0.0017 (7)
C10	0.0375 (9)	0.0284 (8)	0.0483 (10)	-0.0015 (7)	0.0106 (7)	0.0036 (7)
C11	0.0487 (11)	0.0249 (9)	0.0984 (17)	-0.0025 (8)	-0.0175 (11)	0.0050 (9)
C12	0.0367 (8)	0.0364 (9)	0.0391 (9)	-0.0067 (7)	-0.0004 (7)	0.0046 (7)
C13	0.0567 (12)	0.0595 (12)	0.0517 (12)	-0.0214 (10)	0.0102 (10)	0.0122 (10)
C14	0.0356 (8)	0.0316 (9)	0.0529 (11)	0.0045 (7)	0.0065 (8)	-0.0035 (8)
C15	0.0321 (8)	0.0273 (8)	0.0384 (9)	0.0018 (6)	0.0068 (7)	0.0007 (6)
N1	0.0360 (8)	0.0254 (7)	0.0597 (10)	-0.0008 (6)	-0.0008 (7)	0.0048 (6)
N2	0.0368 (7)	0.0309 (7)	0.0370 (7)	-0.0023 (6)	0.0092 (6)	0.0003 (6)
N3	0.0445 (8)	0.0319 (7)	0.0494 (9)	0.0022 (6)	0.0202 (7)	0.0034 (6)
O1	0.1041 (12)	0.0577 (9)	0.0781 (11)	-0.0285 (9)	0.0487 (10)	0.0027 (8)
O2	0.0480 (7)	0.0329 (7)	0.0749 (9)	-0.0048 (5)	0.0318 (7)	-0.0182 (6)
O3	0.0431 (6)	0.0236 (6)	0.0494 (7)	-0.0037 (5)	0.0176 (5)	-0.0026 (5)
O4	0.0732 (9)	0.0259 (6)	0.0739 (9)	0.0032 (6)	0.0383 (7)	0.0049 (6)
O5	0.0638 (9)	0.0543 (8)	0.0784 (10)	0.0023 (7)	0.0471 (8)	0.0018 (7)

Geometric parameters (Å, °)

C1—O1	1.207 (2)	C9—H9B	0.9900
C1—C2	1.460 (2)	C10—C11	1.499 (2)
C1—H1	0.9500	C10—H10A	0.9900
C2—C8	1.374 (2)	C10—H10B	0.9900
C2—C3	1.400 (2)	C11—N1	1.468 (2)
C3—C5	1.371 (2)	C11—H11A	0.9900
C3—H3	0.9500	C11—H11B	0.9900
C4—O2	1.428 (2)	C12—N2	1.315 (2)
C4—H4A	0.9800	C12—N1	1.371 (2)
C4—H4B	0.9800	C12—C13	1.483 (2)
C4—H4C	0.9800	C13—H13A	0.9800
C5—O2	1.3615 (18)	C13—H13B	0.9800
C5—C6	1.412 (2)	C13—H13C	0.9800
C6—O3	1.3567 (18)	C14—C15	1.355 (2)
C6—C7	1.376 (2)	C14—N1	1.356 (2)
C7—C8	1.390 (2)	C14—H14	0.9500
C7—H7	0.9500	C15—N2	1.362 (2)
C8—H8	0.9500	C15—N3	1.428 (2)
C9—O3	1.4304 (19)	N3—O4	1.2235 (17)
C9—C10	1.514 (2)	N3—O5	1.2339 (18)
C9—H9A	0.9900		
O1—C1—C2	125.53 (19)	C9—C10—H10A	109.7
O1—C1—H1	117.2	C11—C10—H10B	109.7

C2—C1—H1	117.2	C9—C10—H10B	109.7
C8—C2—C3	120.47 (14)	H10A—C10—H10B	108.2
C8—C2—C1	118.55 (15)	N1—C11—C10	113.74 (14)
C3—C2—C1	120.98 (16)	N1—C11—H11A	108.8
C5—C3—C2	119.63 (15)	C10—C11—H11A	108.8
C5—C3—H3	120.2	N1—C11—H11B	108.8
C2—C3—H3	120.2	C10—C11—H11B	108.8
O2—C4—H4A	109.5	H11A—C11—H11B	107.7
O2—C4—H4B	109.5	N2—C12—N1	111.65 (15)
H4A—C4—H4B	109.5	N2—C12—C13	125.48 (17)
O2—C4—H4C	109.5	N1—C12—C13	122.86 (16)
H4A—C4—H4C	109.5	C12—C13—H13A	109.5
H4B—C4—H4C	109.5	C12—C13—H13B	109.5
O2—C5—C3	125.59 (15)	H13A—C13—H13B	109.5
O2—C5—C6	114.59 (14)	C12—C13—H13C	109.5
C3—C5—C6	119.82 (14)	H13A—C13—H13C	109.5
O3—C6—C7	125.37 (14)	H13B—C13—H13C	109.5
O3—C6—C5	114.55 (13)	C15—C14—N1	104.31 (15)
C7—C6—C5	120.08 (14)	C15—C14—H14	127.8
C6—C7—C8	119.74 (14)	N1—C14—H14	127.8
C6—C7—H7	120.1	C14—C15—N2	112.96 (14)
C8—C7—H7	120.1	C14—C15—N3	125.07 (15)
C2—C8—C7	120.23 (14)	N2—C15—N3	121.97 (13)
C2—C8—H8	119.9	C14—N1—C12	107.49 (13)
C7—C8—H8	119.9	C14—N1—C11	125.74 (17)
O3—C9—C10	105.75 (12)	C12—N1—C11	126.73 (16)
O3—C9—H9A	110.6	C12—N2—C15	103.59 (13)
C10—C9—H9A	110.6	O4—N3—O5	123.37 (14)
O3—C9—H9B	110.6	O4—N3—C15	119.10 (13)
C10—C9—H9B	110.6	O5—N3—C15	117.52 (13)
H9A—C9—H9B	108.7	C5—O2—C4	116.73 (14)
C11—C10—C9	109.72 (14)	C6—O3—C9	118.76 (12)
C11—C10—H10A	109.7		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4B \cdots O4 ⁱ	0.98	2.58	3.415 (3)	144
C8—H8 \cdots O4 ⁱⁱ	0.95	2.51	3.229 (2)	133
C10—H10B \cdots O2 ⁱⁱⁱ	0.99	2.56	3.312 (2)	133
C10—H10A \cdots O1 ^{iv}	0.99	2.58	3.461 (2)	148
C14—H14 \cdots O1 ^{iv}	0.95	2.29	3.166 (2)	153

Symmetry codes: (i) $x+1/2, -y+1/2, z-1/2$; (ii) $x, y+1, z$; (iii) $-x, -y+1, -z$; (iv) $x-1/2, -y+3/2, z+1/2$.

Fig. 1

