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2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)-ethyl 3-bromobenzoate

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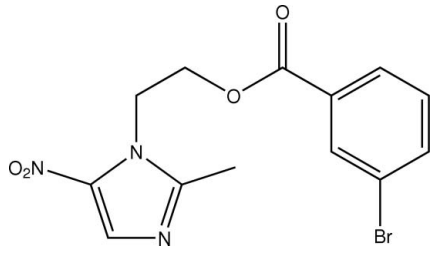
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.014 Å; R factor = 0.061; wR factor = 0.119; data-to-parameter ratio = 14.0.

The molecule of the title compound, $C_{13}H_{12}BrN_3O_4$, is non-planar, as indicated in the dihedral angle of 59.5 (4)° formed between the least-squares planes through the imidazole and benzene rings. In the crystal, molecules are connected *via* C—H···O contacts, forming a supramolecular chain.

Related literature

For potential pharmacological uses of benzoic acid derivatives, see: Correa-Basurto *et al.* (2005); Jetten *et al.* (1987); Kelly *et al.* (2007); Sato *et al.* (2005). For a related structure, see: Wang *et al.* (2008).



Experimental

Crystal data

$C_{13}H_{12}BrN_3O_4$
M_r = 354.17
Monoclinic, *Cc*
a = 11.871 (2) Å
b = 19.840 (4) Å
c = 7.1983 (13) Å
β = 124.488 (3)°
V = 1397.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.96 mm^{-1}
T = 150 K
0.20 × 0.07 × 0.03 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.589, T_{max} = 0.916
5456 measured reflections
2680 independent reflections
1636 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.119
S = 0.86
2680 reflections
191 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}
Absolute structure: Flack (1983), 1301 Friedel pairs
Flack parameter: 0.091 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4a···O2^{i}	0.99	2.59	3.494 (10)	152

Symmetry code: (i) $x, y, z-1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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: TK2425).

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supporting information

Acta Cryst. (2009). E**65**, o1176 [doi:10.1107/S1600536809015499]

2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)ethyl 3-bromobenzoate

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S1. Comment

Derivatives of benzoic acid offer promise as compounds that possess multifunctional physiological activity (hypolesterolemic, antitumor, antithrombic, *etc*.) and do not cause hypervitaminosis and other side-effects (Jetten *et al.* 1987). It has been reported that synthesized benzoic acid derivatives of the amide- and chalcone-series show inhibitory activity of squamous cell differentiation of rabbit traeheal epithelial cell but induce differentiation of mouse embryonal carcinoma F9 and human promyelocytic leukemia HL60 cells (Correa-Basurto *et al.*, 2005). *p*-Aminobenzoic acid derivatives were evaluated as acetylcholinesterase inhibitors (AChEIs) (Sato *et al.*, 2005). Benzoic acid derivatives have also been found to exhibit cytotoxic effects on the MDA-MB-435-S—F breast cancer cell line (Kelly *et al.*, 2007).

In the crystal structure of the title compound (I), Fig. 1, the key C=O and C—N bond distances are in agreement with those observed in the related structure of imidazolmethyl phthalimide (Wang *et al.*, 2008).

S2. Experimental

Metronidazole (5 g, 29.23 mmol) was added to 3-bromobenzoic acid (7.64 g, 38.01 mmol) dissolved in anhydrous CH_2Cl_2 (10 ml). Then 4-dimethylaminopyridine (0.15 equiv.) and dicyclohexylcarbodiimide (1.25 equiv) were added, and the resulting solution stirred. After 12 h, the solvent was evaporated under reduced pressure. The crude reaction mixture was subjected to flash column chromatography over silica gel, successively eluting with n-hexane–ethyl acetate (3:7) which afforded (I) in 70% yield. Colorless crystals were obtained from the slow evaporation of a CH_2Cl_2 solution of (I).

S3. Refinement

H atoms were placed in calculated positions, C—H = 0.95–0.99 Å, and included in the riding model approximation with U_{iso} set to $1.5U_{eq}$(C) for methyl-H atoms and $1.2U_{eq}$(C) for remaining H atoms.

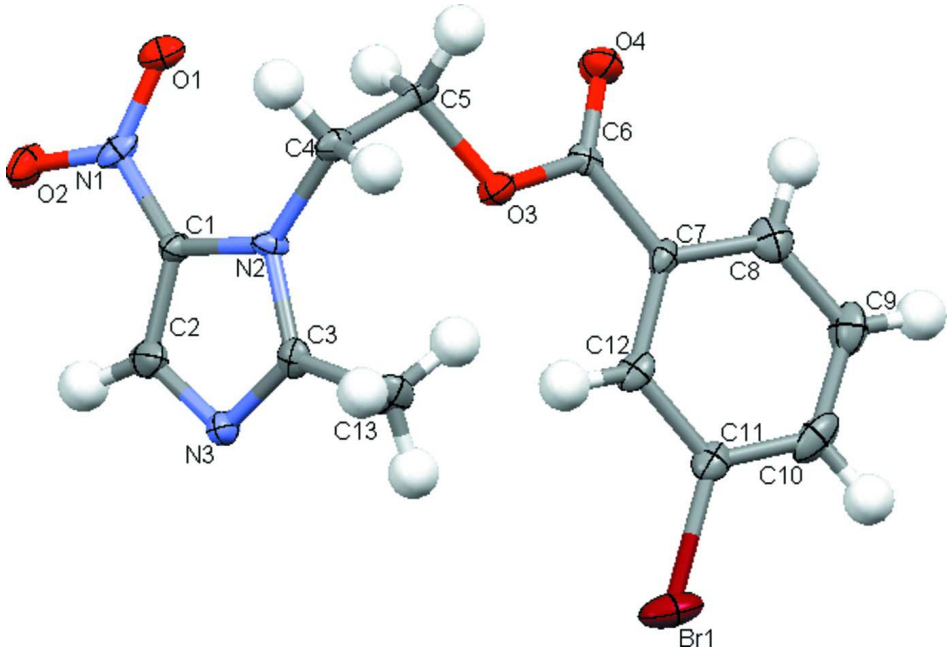

Figure 1

Molecular Structure of (I) show atom labelling and 30% displacement ellipsoids.

2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)ethyl 3-bromobenzoate

Crystal data

$C_{13}H_{12}BrN_3O_4$
$M_r = 354.17$
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Hall symbol: C -2yc
$a = 11.871\ (2)$ Å
$b = 19.840\ (4)$ Å
$c = 7.1983\ (13)$ Å
$\beta = 124.488\ (3)°$
$V = 1397.4\ (4)$ Å^3
$Z = 4$

$F(000) = 712$
$D_x = 1.683$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 616 reflections
$\theta = 3.7–23.3°$
$\mu = 2.96$ mm^{-1}
$T = 150$ K
Needle, colourless
0.20 × 0.07 × 0.03 mm

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Radiation source: fine-focus sealed tube
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φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.589$, $T_{max} = 0.916$

5456 measured reflections
2680 independent reflections
1636 reflections with $I > 2\sigma(I)$
$R_{int} = 0.087$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.1°$
$h = -14\rightarrow14$
$k = -24\rightarrow23$
$l = -8\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.119$
$S = 0.86$

2680 reflections
191 parameters
2 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.66$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}
Absolute structure: Flack (1983), 1301 Friedel pairs
Absolute structure parameter: 0.091 (17)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.81001 (10)	0.59782 (5)	0.72927 (13)	0.0588 (3)
O1	0.0138 (5)	0.6343 (3)	0.5323 (9)	0.0363 (14)
O2	0.0896 (5)	0.7074 (3)	0.8019 (9)	0.0434 (15)
O3	0.2933 (5)	0.5475 (3)	0.4184 (9)	0.0309 (14)
O4	0.2623 (6)	0.4375 (3)	0.4381 (11)	0.0437 (16)
N1	0.0966 (6)	0.6786 (3)	0.6597 (11)	0.0322 (16)
N2	0.2152 (6)	0.6769 (3)	0.4669 (9)	0.0234 (14)
N3	0.3904 (6)	0.7449 (3)	0.6962 (11)	0.0324 (16)
C1	0.2023 (8)	0.6976 (4)	0.6362 (12)	0.0261 (17)
C2	0.3074 (10)	0.7391 (4)	0.7650 (18)	0.042 (3)
H2	0.3216	0.7622	0.8924	0.050*
C3	0.3307 (8)	0.7090 (4)	0.5099 (13)	0.0281 (18)
C4	0.1261 (7)	0.6329 (4)	0.2730 (12)	0.0280 (18)
H4A	0.1413	0.6422	0.1537	0.034*
H4B	0.0295	0.6436	0.2115	0.034*
C5	0.1513 (7)	0.5597 (4)	0.3332 (13)	0.0294 (19)
H5A	0.1344	0.5490	0.4498	0.035*
H5B	0.0903	0.5315	0.1988	0.035*
C6	0.3328 (8)	0.4832 (4)	0.4592 (12)	0.0222 (18)
C7	0.4765 (8)	0.4759 (4)	0.5349 (12)	0.0263 (19)
C8	0.5272 (9)	0.4134 (4)	0.5409 (13)	0.041 (2)
H8	0.4686	0.3754	0.4966	0.049*
C9	0.6614 (10)	0.4036 (5)	0.6098 (14)	0.048 (2)
H9	0.6962	0.3596	0.6198	0.057*
C10	0.7422 (9)	0.4597 (5)	0.6630 (13)	0.049 (3)
H10	0.8333	0.4538	0.7053	0.059*
C11	0.6978 (8)	0.5231 (4)	0.6579 (12)	0.035 (2)
C12	0.5625 (7)	0.5325 (4)	0.5850 (12)	0.032 (2)
H12	0.5277	0.5768	0.5688	0.038*

C13	0.3796 (8)	0.7029 (4)	0.3597 (13)	0.043 (2)
H13A	0.3319	0.7359	0.2373	0.064*
H13B	0.3608	0.6573	0.2962	0.064*
H13C	0.4781	0.7115	0.4472	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0321 (4)	0.0955 (8)	0.0384 (5)	−0.0218 (7)	0.0137 (4)	0.0017 (7)
O1	0.025 (3)	0.046 (4)	0.035 (3)	−0.002 (3)	0.014 (3)	0.006 (3)
O2	0.038 (3)	0.065 (4)	0.039 (3)	0.001 (3)	0.028 (3)	−0.007 (3)
O3	0.025 (3)	0.031 (3)	0.036 (4)	0.002 (3)	0.017 (3)	0.005 (3)
O4	0.042 (4)	0.031 (3)	0.062 (4)	−0.003 (3)	0.032 (4)	0.005 (3)
N1	0.021 (4)	0.044 (4)	0.030 (4)	0.007 (3)	0.014 (3)	0.013 (3)
N2	0.025 (4)	0.028 (4)	0.018 (3)	−0.009 (3)	0.012 (3)	−0.003 (3)
N3	0.031 (4)	0.036 (4)	0.036 (4)	−0.007 (3)	0.022 (4)	−0.012 (3)
C1	0.024 (4)	0.031 (4)	0.025 (4)	−0.005 (4)	0.015 (4)	−0.006 (4)
C2	0.030 (5)	0.030 (5)	0.059 (7)	0.003 (5)	0.021 (5)	0.000 (5)
C3	0.035 (5)	0.024 (4)	0.030 (5)	0.006 (4)	0.021 (4)	0.008 (4)
C4	0.027 (4)	0.039 (5)	0.022 (4)	−0.005 (4)	0.017 (4)	0.000 (4)
C5	0.021 (4)	0.043 (5)	0.028 (5)	−0.003 (4)	0.017 (4)	0.008 (4)
C6	0.027 (5)	0.028 (5)	0.010 (4)	−0.004 (4)	0.010 (3)	−0.002 (4)
C7	0.024 (4)	0.037 (5)	0.019 (4)	0.005 (4)	0.013 (4)	−0.002 (4)
C8	0.044 (6)	0.046 (6)	0.024 (5)	0.004 (5)	0.014 (4)	0.003 (4)
C9	0.049 (6)	0.049 (6)	0.041 (6)	0.022 (5)	0.023 (5)	0.011 (5)
C10	0.038 (5)	0.085 (8)	0.035 (6)	0.012 (5)	0.027 (5)	0.012 (5)
C11	0.029 (5)	0.050 (6)	0.021 (4)	0.005 (4)	0.011 (4)	0.000 (4)
C12	0.025 (4)	0.045 (5)	0.021 (4)	0.009 (4)	0.010 (4)	0.009 (4)
C13	0.053 (6)	0.043 (5)	0.044 (5)	−0.010 (5)	0.035 (5)	0.006 (4)

Geometric parameters (Å, º)

Br1—C11	1.861 (8)	C4—H4B	0.9900
O1—N1	1.248 (7)	C5—H5A	0.9900
O2—N1	1.216 (8)	C5—H5B	0.9900
O3—C6	1.334 (9)	C6—C7	1.475 (10)
O3—C5	1.451 (8)	C7—C8	1.369 (10)
O4—C6	1.183 (9)	C7—C12	1.420 (11)
N1—C1	1.410 (9)	C8—C9	1.389 (12)
N2—C1	1.374 (9)	C8—H8	0.9500
N2—C3	1.378 (9)	C9—C10	1.375 (11)
N2—C4	1.468 (8)	C9—H9	0.9500
N3—C3	1.317 (9)	C10—C11	1.356 (11)
N3—C2	1.334 (11)	C10—H10	0.9500
C1—C2	1.336 (12)	C11—C12	1.391 (10)
C2—H2	0.9500	C12—H12	0.9500
C3—C13	1.493 (10)	C13—H13A	0.9800
C4—C5	1.497 (10)	C13—H13B	0.9800

C4—H4A	0.9900	C13—H13C	0.9800
C6—O3—C5	115.6 (6)	H5A—C5—H5B	108.7
O2—N1—O1	123.3 (6)	O4—C6—O3	124.8 (7)
O2—N1—C1	117.7 (7)	O4—C6—C7	124.0 (8)
O1—N1—C1	119.0 (6)	O3—C6—C7	111.3 (7)
C1—N2—C3	105.0 (6)	C8—C7—C12	118.0 (7)
C1—N2—C4	129.7 (6)	C8—C7—C6	119.7 (8)
C3—N2—C4	125.2 (6)	C12—C7—C6	122.1 (7)
C3—N3—C2	104.3 (7)	C7—C8—C9	122.2 (8)
C2—C1—N2	105.7 (7)	C7—C8—H8	118.9
C2—C1—N1	128.8 (8)	C9—C8—H8	118.9
N2—C1—N1	125.4 (6)	C10—C9—C8	117.7 (8)
N3—C2—C1	113.0 (9)	C10—C9—H9	121.1
N3—C2—H2	123.5	C8—C9—H9	121.1
C1—C2—H2	123.5	C11—C10—C9	122.9 (8)
N3—C3—N2	111.8 (6)	C11—C10—H10	118.5
N3—C3—C13	125.1 (7)	C9—C10—H10	118.5
N2—C3—C13	123.0 (7)	C10—C11—C12	118.9 (8)
N2—C4—C5	112.5 (6)	C10—C11—Br1	121.6 (7)
N2—C4—H4A	109.1	C12—C11—Br1	119.3 (6)
C5—C4—H4A	109.1	C11—C12—C7	120.0 (8)
N2—C4—H4B	109.1	C11—C12—H12	120.0
C5—C4—H4B	109.1	C7—C12—H12	120.0
H4A—C4—H4B	107.8	C3—C13—H13A	109.5
O3—C5—C4	106.1 (6)	C3—C13—H13B	109.5
O3—C5—H5A	110.5	H13A—C13—H13B	109.5
C4—C5—H5A	110.5	C3—C13—H13C	109.5
O3—C5—H5B	110.5	H13A—C13—H13C	109.5
C4—C5—H5B	110.5	H13B—C13—H13C	109.5
C3—N2—C1—C2	−0.1 (8)	C6—O3—C5—C4	−173.1 (6)
C4—N2—C1—C2	−177.6 (7)	N2—C4—C5—O3	−59.7 (7)
C3—N2—C1—N1	178.8 (7)	C5—O3—C6—O4	−2.7 (11)
C4—N2—C1—N1	1.3 (12)	C5—O3—C6—C7	177.4 (6)
O2—N1—C1—C2	7.5 (12)	O4—C6—C7—C8	13.3 (12)
O1—N1—C1—C2	−173.8 (8)	O3—C6—C7—C8	−166.7 (7)
O2—N1—C1—N2	−171.2 (7)	O4—C6—C7—C12	−172.0 (7)
O1—N1—C1—N2	7.5 (10)	O3—C6—C7—C12	8.0 (10)
C3—N3—C2—C1	3.8 (10)	C12—C7—C8—C9	4.8 (12)
N2—C1—C2—N3	−2.3 (10)	C6—C7—C8—C9	179.8 (7)
N1—C1—C2—N3	178.8 (7)	C7—C8—C9—C10	−3.0 (13)
C2—N3—C3—N2	−3.8 (9)	C8—C9—C10—C11	2.2 (13)
C2—N3—C3—C13	176.0 (8)	C9—C10—C11—C12	−3.3 (13)
C1—N2—C3—N3	2.5 (8)	C9—C10—C11—Br1	−179.6 (6)
C4—N2—C3—N3	−179.8 (6)	C10—C11—C12—C7	5.1 (11)
C1—N2—C3—C13	−177.3 (7)	Br1—C11—C12—C7	−178.5 (6)
C4—N2—C3—C13	0.3 (11)	C8—C7—C12—C11	−5.8 (11)

C1—N2—C4—C5	−81.4 (9)	C6—C7—C12—C11	179.4 (7)
C3—N2—C4—C5	101.6 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4a···O2^{i}	0.99	2.59	3.494 (10)	152

Symmetry code: (i) *x*, *y*, *z*−1.