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2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)-ethyl *N*-methylcarbamate

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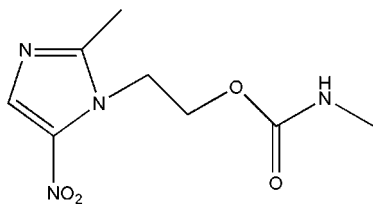
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.031; wR factor = 0.089; data-to-parameter ratio = 13.2.

In the title compound, $\text{C}_8\text{H}_{12}\text{N}_4\text{O}_4$, the essentially planar methylcarbamoyloxymethyl group [maximum deviation 0.038 (3) Å] and the imidazole ring make a dihedral angle of 48.47 (3)°. The crystal packing is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, which link the molecules into infinite ribbons running along the a axis, and by weak $\pi-\pi$ stacking interactions [centroid-centroid distance = 3.894 (2) Å].

Related literature

For biological activity, see: Cina *et al.* (1996); Karamanakos *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_8\text{H}_{12}\text{N}_4\text{O}_4$

$M_r = 228.22$

Monoclinic, $P2_1/n$
 $a = 9.6959$ (12) Å
 $b = 7.2898$ (9) Å
 $c = 15.589$ (2) Å
 $\beta = 101.400$ (2)°
 $V = 1080.1$ (2) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 273$ (2) K
 $0.15 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: none
 5515 measured reflections

1921 independent reflections
 1622 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.089$
 $S = 1.05$
 1921 reflections

146 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N4}-\text{H4}\cdots\text{N1}^{\text{i}}$	0.86	2.20	3.0416 (17)	165
$\text{C2}-\text{H2}\cdots\text{O4}^{\text{ii}}$	0.93	2.34	3.2492 (18)	166
$\text{C8}-\text{H8B}\cdots\text{O4}^{\text{iii}}$	0.96	2.57	3.498 (2)	164

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2452).

References

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supplementary materials

Acta Cryst. (2009). E65, o116 [doi:10.1107/S1600536808041676]

2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)ethyl *N*-methylcarbamate

G.-Y. Duan, C.-C. Xia and Y.-L. Xiao

Comment

2-(2-methyl-5-nitro-1*H*-imidazol-1-yl)ethanol is an anti-anaerobic bacteria and anti-infusorium agent to treatment of infection which is called metronidazole (Cina *et al.*, 1996; Karamanakos *et al.*, 2007). The title compound is a derivative of it. In this paper, we report the crystal structure of the title compound.

In (I) (Fig. 1), all bond lengths are normal (Allen *et al.*, 1987). Atoms C4, C5, N3, O1, O2 lie in the plane of the imidazole ring (C1/C2/C3/N1/N2) with maximum deviations 0.004 (2)Å for O1. The essentially planar methyl methylcarbamate moiety (C6—C8/N4/O3/O4) and the imidazole ring make a dihedral angle of 48.47 (3)°. The relatively short distance of 3.894 (2)Å between the centroids of imidazole ring C1/C2/C3/N1/N2 [at (1-*X*, -*Y*, 1-*Z*)] indicates the presence of weak π - π interactions, which contribute to the stability of the crystal packing. The crystal packing is also stabilized by intermolecular N—H \cdots N and C—H \cdots O hydrogen bonds,

Experimental

The title compound was prepared by reaction of 1.71 g (0.01 mol) 2-(2-methyl-5-nitro-1*H*-imidazol-1-yl)ethanol with 0.68 g (0.12 mol) methyl isocyanate catalyzed by 1 g triethylamine in 20 ml toluene at room temperature, yield 86.4%. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a metanol solution at room temperature for two weeks.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, N—H = 0.86 Å, and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2$ (1.5 for methyl) times $U_{\text{eq}}(\text{C}, \text{N})$.

Figures

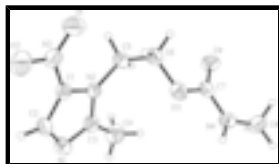


Fig. 1. View of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

2-(2-Methyl-5-nitro-1*H*-imidazol-1-yl)ethyl *N*-methylcarbamate

Crystal data

C₈H₁₂N₄O₄

$M_r = 228.22$

$F_{000} = 480$

$D_x = 1.403 \text{ Mg m}^{-3}$

supplementary materials

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 9.6959$ (12) Å

$b = 7.2898$ (9) Å

$c = 15.589$ (2) Å

$\beta = 101.400$ (2)°

$V = 1080.1$ (2) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2505 reflections

$\theta = 2.7$ – 27.1 °

$\mu = 0.11$ mm⁻¹

$T = 273$ (2) K

Block, yellow

$0.15 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 273$ (2) K

φ and ω scans

Absorption correction: none

5515 measured reflections

1921 independent reflections

1622 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.3$ °

$h = -11 \rightarrow 11$

$k = -8 \rightarrow 8$

$l = -9 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.089$

$S = 1.05$

1921 reflections

146 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2 + 0.2462P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.19$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Extinction correction: SHELXTL (Sheldrick, 2008),

$$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.011 (2)

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	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.41899 (13)	0.31179 (19)	0.60227 (7)	0.0673 (4)
O2	0.62309 (12)	0.35345 (19)	0.56977 (8)	0.0693 (4)
O3	0.06913 (10)	0.38773 (15)	0.39251 (7)	0.0495 (3)
O4	-0.13394 (10)	0.24805 (16)	0.40569 (7)	0.0516 (3)
N1	0.45446 (13)	0.11166 (19)	0.33518 (8)	0.0511 (3)
N2	0.32310 (11)	0.14897 (15)	0.43564 (7)	0.0368 (3)
N3	0.50075 (13)	0.29867 (17)	0.55195 (8)	0.0461 (3)
N4	-0.11627 (13)	0.41147 (18)	0.28483 (8)	0.0489 (3)
H4	-0.0602	0.4739	0.2598	0.059*
C1	0.45604 (14)	0.21818 (18)	0.46825 (9)	0.0374 (3)
C2	0.53355 (15)	0.1938 (2)	0.40604 (10)	0.0456 (4)
H2	0.6272	0.2283	0.4111	0.055*
C3	0.32821 (15)	0.0863 (2)	0.35445 (9)	0.0437 (4)
C4	0.20850 (19)	-0.0011 (3)	0.29424 (12)	0.0657 (5)
H4A	0.2356	-0.0287	0.2397	0.099*
H4B	0.1297	0.0812	0.2841	0.099*
H4C	0.1828	-0.1125	0.3200	0.099*
C5	0.20058 (15)	0.1428 (2)	0.47777 (10)	0.0453 (4)
H5A	0.2301	0.0991	0.5373	0.054*
H5B	0.1326	0.0562	0.4467	0.054*
C6	0.13134 (15)	0.3268 (2)	0.47920 (10)	0.0489 (4)
H6A	0.0591	0.3191	0.5141	0.059*
H6B	0.2007	0.4159	0.5064	0.059*
C7	-0.06789 (14)	0.34081 (19)	0.36319 (9)	0.0390 (3)
C8	-0.26021 (17)	0.3868 (2)	0.24023 (11)	0.0564 (4)
H8A	-0.2734	0.2632	0.2190	0.085*
H8B	-0.2811	0.4705	0.1918	0.085*
H8C	-0.3219	0.4104	0.2801	0.085*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0660 (8)	0.0934 (10)	0.0442 (6)	-0.0052 (7)	0.0153 (6)	-0.0090 (6)
O2	0.0506 (7)	0.0844 (9)	0.0671 (8)	-0.0186 (6)	-0.0026 (6)	-0.0062 (7)
O3	0.0351 (5)	0.0590 (7)	0.0568 (7)	-0.0004 (5)	0.0146 (5)	0.0064 (5)
O4	0.0431 (6)	0.0645 (7)	0.0486 (6)	-0.0100 (5)	0.0123 (5)	0.0118 (5)
N1	0.0494 (7)	0.0567 (8)	0.0504 (8)	0.0087 (6)	0.0176 (6)	-0.0046 (6)
N2	0.0346 (6)	0.0373 (6)	0.0394 (6)	0.0021 (5)	0.0091 (5)	0.0027 (5)
N3	0.0447 (7)	0.0477 (7)	0.0436 (7)	-0.0008 (6)	0.0029 (6)	0.0050 (6)
N4	0.0441 (7)	0.0564 (8)	0.0491 (7)	-0.0010 (6)	0.0162 (6)	0.0130 (6)
C1	0.0350 (7)	0.0356 (7)	0.0410 (7)	0.0018 (6)	0.0062 (6)	0.0040 (6)
C2	0.0364 (8)	0.0477 (8)	0.0547 (9)	0.0043 (6)	0.0141 (7)	0.0042 (7)

supplementary materials

C3	0.0459 (8)	0.0405 (8)	0.0439 (8)	0.0073 (6)	0.0071 (6)	-0.0020 (6)
C4	0.0633 (11)	0.0710 (12)	0.0582 (10)	-0.0002 (9)	0.0007 (8)	-0.0172 (9)
C5	0.0378 (7)	0.0522 (9)	0.0481 (8)	-0.0052 (6)	0.0138 (6)	0.0045 (7)
C6	0.0361 (8)	0.0641 (10)	0.0478 (9)	0.0024 (7)	0.0112 (6)	-0.0061 (7)
C7	0.0359 (7)	0.0385 (7)	0.0460 (8)	0.0023 (6)	0.0162 (6)	-0.0017 (6)
C8	0.0554 (10)	0.0609 (10)	0.0512 (9)	-0.0011 (8)	0.0062 (7)	0.0107 (8)

Geometric parameters (Å, °)

O1—N3	1.2241 (16)	C1—C2	1.3509 (19)
O2—N3	1.2303 (16)	C2—H2	0.9300
O3—C7	1.3605 (17)	C3—C4	1.485 (2)
O3—C6	1.4370 (18)	C4—H4A	0.9600
O4—C7	1.2140 (16)	C4—H4B	0.9600
N1—C3	1.3297 (19)	C4—H4C	0.9600
N1—C2	1.354 (2)	C5—C6	1.503 (2)
N2—C3	1.3555 (18)	C5—H5A	0.9700
N2—C1	1.3840 (17)	C5—H5B	0.9700
N2—C5	1.4673 (17)	C6—H6A	0.9700
N3—C1	1.4181 (18)	C6—H6B	0.9700
N4—C7	1.3233 (19)	C8—H8A	0.9600
N4—C8	1.442 (2)	C8—H8B	0.9600
N4—H4	0.8600	C8—H8C	0.9600
C7—O3—C6	116.02 (11)	H4A—C4—H4C	109.5
C3—N1—C2	105.99 (12)	H4B—C4—H4C	109.5
C3—N2—C1	105.22 (11)	N2—C5—C6	112.52 (12)
C3—N2—C5	126.14 (12)	N2—C5—H5A	109.1
C1—N2—C5	128.64 (11)	C6—C5—H5A	109.1
O1—N3—O2	123.20 (13)	N2—C5—H5B	109.1
O1—N3—C1	120.20 (12)	C6—C5—H5B	109.1
O2—N3—C1	116.60 (13)	H5A—C5—H5B	107.8
C7—N4—C8	121.95 (12)	O3—C6—C5	111.56 (12)
C7—N4—H4	119.0	O3—C6—H6A	109.3
C8—N4—H4	119.0	C5—C6—H6A	109.3
C2—C1—N2	107.35 (12)	O3—C6—H6B	109.3
C2—C1—N3	127.06 (13)	C5—C6—H6B	109.3
N2—C1—N3	125.59 (12)	H6A—C6—H6B	108.0
C1—C2—N1	109.76 (13)	O4—C7—N4	126.27 (13)
C1—C2—H2	125.1	O4—C7—O3	122.83 (13)
N1—C2—H2	125.1	N4—C7—O3	110.89 (12)
N1—C3—N2	111.67 (13)	N4—C8—H8A	109.5
N1—C3—C4	123.81 (14)	N4—C8—H8B	109.5
N2—C3—C4	124.52 (14)	H8A—C8—H8B	109.5
C3—C4—H4A	109.5	N4—C8—H8C	109.5
C3—C4—H4B	109.5	H8A—C8—H8C	109.5
H4A—C4—H4B	109.5	H8B—C8—H8C	109.5
C3—C4—H4C	109.5		
C3—N2—C1—C2	-0.11 (15)	C1—N2—C3—N1	0.19 (16)
C5—N2—C1—C2	-179.97 (13)	C5—N2—C3—N1	-179.95 (13)

C3—N2—C1—N3	-179.87 (13)	C1—N2—C3—C4	179.78 (15)
C5—N2—C1—N3	0.3 (2)	C5—N2—C3—C4	-0.4 (2)
O1—N3—C1—C2	-179.83 (15)	C3—N2—C5—C6	-104.57 (16)
O2—N3—C1—C2	0.5 (2)	C1—N2—C5—C6	75.25 (18)
O1—N3—C1—N2	-0.1 (2)	C7—O3—C6—C5	91.29 (14)
O2—N3—C1—N2	-179.77 (13)	N2—C5—C6—O3	66.34 (16)
N2—C1—C2—N1	0.01 (16)	C8—N4—C7—O4	1.1 (2)
N3—C1—C2—N1	179.76 (13)	C8—N4—C7—O3	-178.06 (13)
C3—N1—C2—C1	0.11 (17)	C6—O3—C7—O4	-2.3 (2)
C2—N1—C3—N2	-0.19 (17)	C6—O3—C7—N4	176.83 (12)
C2—N1—C3—C4	-179.78 (15)		

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>
N4—H4...N1 ⁱ	0.86	2.20	3.0416 (17)	165
C2—H2...O4 ⁱⁱ	0.93	2.34	3.2492 (18)	166
C8—H8B...O4 ⁱⁱⁱ	0.96	2.57	3.498 (2)	164

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

Fig. 1

