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Ethyl 2-(2-methyl-4-nitro-1*H*-imidazol-1-yl)acetate

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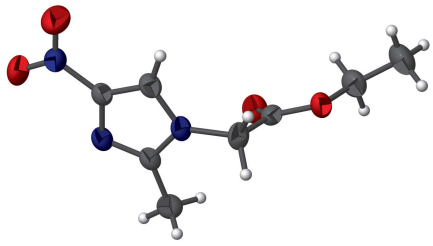
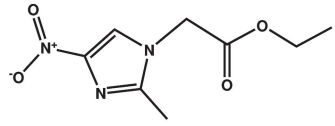
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_8H_{11}N_3O_4$, the imidazole ring and the nitro group are nearly coplanar, with the largest deviation from the mean plane being 0.119 (2) Å. The mean plane through the acetate group is approximately perpendicular to the imidazole ring, subtending a dihedral angle of 75.71 (13)°. In the crystal, molecules are linked by weak C—H···O and very weak C—H···N hydrogen bonds, forming a three-dimensional network. There is also a weak C—H···π(imidazole) interaction, which contributes to the stability of the crystal packing arrangement.

3D view



Chemical scheme



Structure description

Imidazoles are an important class of heterocyclic compounds that are abundant in the structures of many natural and synthetic pharmacologically active substances (Neildé *et al.*, 2014; Adamovich *et al.*, 2014). Some nitroimidazole derivatives have been identified as notable radiosensitizers, antiprotozoal, antifungal and antibacterial or anti-epileptic agents (Olender *et al.*, 2009; Duan *et al.*, 2014; Sutherland *et al.*, 2010).

The molecule of the title compound is build up from a nitro- and methyl-substituted imidazole ring (C1–C3/N2/N3) linked to an ethylacetate moiety, as shown in Fig. 1. The nitro group and the imidazole ring are coplanar with a maximum deviation from the mean plane of 0.119 (2) Å for O1. The imidazole ring makes a dihedral angle of 75.71 (13)° with the plane through the acetate group. The title compound is achiral, although it crystallizes in a chiral space group.

The crystal structure cohesion is ensured by C—H···O and C—H···N hydrogen-bonding interactions (Table 1). There is also a weak C5—H5*A*···π interaction, which contributes to the stability of the crystal packing arrangement, as shown in Fig. 2.

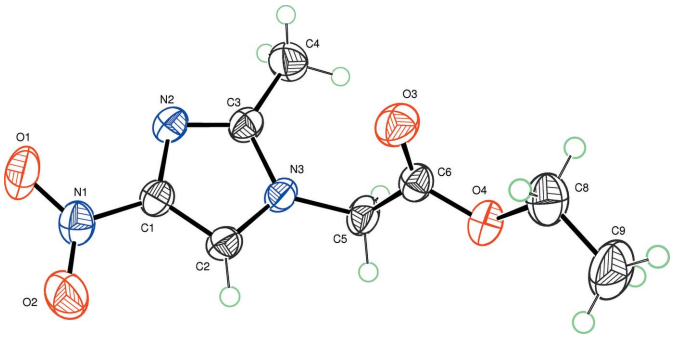


Figure 1
Plot of the molecule of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

Synthesis and crystallization

To a solution of 2-methyl-5-nitro-1*H*-imidazole (7.87 mmol) in DMSO was added potassium hydroxide (8.7 mmol). After 15 min of stirring at 298 K, ethyl bromoacetate (15.74 mmol) was added dropwise. Upon disappearance of the starting material as indicated by TLC, the mixture was added to ice–water and extracted with ethyl acetate. The organic phase was washed with brine and dried over magnesium sulfate. The solvent was evaporated *in vacuo*. The resulting residue was purified by column chromatography (EtOAc/hexane 09/01). The title compound was recrystallized from ethanol at room temperature giving colourless crystals (m.p. 351 K, yield 68%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements and the University Sultan Moulay Slimane, Beni-Mellal, Morocco, for financial support.

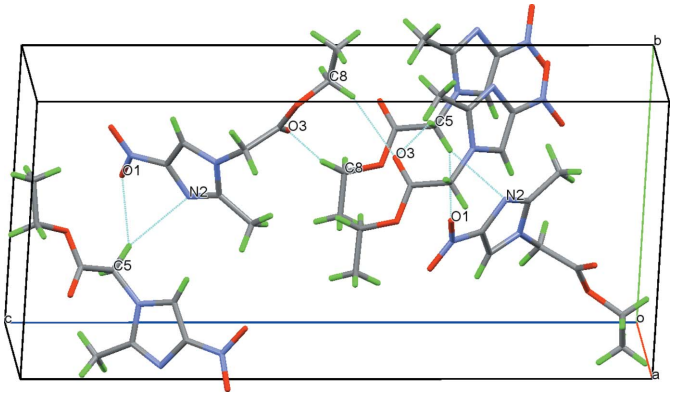


Figure 2
The crystal packing for the title compound, showing hydrogen bonds as dashed lines.

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

Cg is the centroid of the imidazole ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O3^{i}	0.97	2.43	3.224 (2)	139
C8—H8*B*···O3^{ii}	0.97	2.54	3.446 (3)	156
C5—H5*B*···N2^{iii}	0.97	2.65	3.586 (2)	161
C5—H5*B*···O1^{iii}	0.97	2.62	3.387 (2)	136
C5—H5*A*···*Cg*^{i}	0.97	2.98	3.651 (2)	128

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{11}N_3O_4$
M_r	213.20
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, *b*, *c* (Å)	4.4793 (2), 10.3596 (5), 21.5724 (11)
V (Å^3)	1001.04 (8)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.12
Crystal size (mm)	0.13 × 0.12 × 0.10
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.635, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23416, 2802, 2488
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.694
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.037, 0.100, 1.06
No. of reflections	2802
No. of parameters	137
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.15

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

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full crystallographic data

IUCrData (2016). **1**, x160588 [doi:10.1107/S2414314616005885]

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Ethyl 2-(2-methyl-4-nitro-1*H*-imidazol-1-yl)acetate

Crystal data

$C_8H_{11}N_3O_4$
M_r = 213.20
Orthorhombic, $P2_12_12_1$
a = 4.4793 (2) Å
b = 10.3596 (5) Å
c = 21.5724 (11) Å
V = 1001.04 (8) Å^3
Z = 4
F(000) = 448
D_x = 1.415 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2802 reflections
θ = 2.2–29.6°
μ = 0.12 mm^{-1}
T = 296 K
Block, colourless
0.13 × 0.12 × 0.10 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.635, T_{max} = 0.746
23416 measured reflections
2802 independent reflections
2488 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 29.6°, θ_{min} = 2.2°
h = −6→6
k = −11→14
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.100
S = 1.06
2802 reflections
137 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.0658P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6714 (4)	0.89444 (15)	0.23607 (7)	0.0340 (3)
C2	0.8179 (4)	0.78660 (15)	0.25496 (7)	0.0357 (4)
H2	0.8107	0.7045	0.2376	0.043*
C3	0.9159 (4)	0.95500 (14)	0.31441 (8)	0.0351 (4)
C4	1.0510 (5)	1.03126 (19)	0.36538 (9)	0.0512 (5)
H4A	1.0123	0.9894	0.4043	0.077*
H4B	0.9655	1.1162	0.3658	0.077*
H4C	1.2626	1.0375	0.3590	0.077*
C5	1.1594 (4)	0.74202 (18)	0.34365 (8)	0.0395 (4)
H5A	1.3374	0.7879	0.3567	0.047*
H5B	1.2214	0.6676	0.3196	0.047*
C6	0.9893 (4)	0.69707 (15)	0.40023 (8)	0.0346 (3)
C8	0.9589 (6)	0.53412 (19)	0.47602 (9)	0.0541 (5)
H8A	0.7444	0.5356	0.4698	0.065*
H8B	1.0053	0.5821	0.5134	0.065*
C9	1.0674 (7)	0.3982 (2)	0.48146 (12)	0.0703 (7)
H9A	0.9722	0.3572	0.5161	0.105*
H9B	1.0201	0.3519	0.4442	0.105*
H9C	1.2797	0.3982	0.4876	0.105*
N1	0.4717 (4)	0.90250 (14)	0.18451 (7)	0.0422 (3)
N2	0.7284 (4)	0.99894 (13)	0.27239 (6)	0.0371 (3)
N3	0.9776 (3)	0.82629 (12)	0.30513 (6)	0.0338 (3)
O1	0.3344 (4)	1.00273 (14)	0.17624 (8)	0.0603 (4)
O2	0.4468 (5)	0.80706 (15)	0.15122 (7)	0.0671 (5)
O3	0.7749 (3)	0.75150 (13)	0.42096 (6)	0.0492 (3)
O4	1.1105 (3)	0.59101 (12)	0.42288 (6)	0.0421 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0341 (8)	0.0307 (7)	0.0370 (7)	0.0001 (7)	0.0003 (7)	0.0044 (6)
C2	0.0422 (9)	0.0287 (7)	0.0364 (7)	0.0035 (7)	0.0022 (7)	0.0001 (6)
C3	0.0386 (9)	0.0282 (7)	0.0385 (8)	0.0003 (7)	0.0017 (7)	0.0029 (6)
C4	0.0637 (13)	0.0421 (9)	0.0479 (9)	−0.0055 (10)	−0.0087 (10)	−0.0026 (8)
C5	0.0324 (8)	0.0418 (8)	0.0443 (8)	0.0106 (7)	−0.0010 (7)	0.0077 (7)
C6	0.0312 (8)	0.0336 (7)	0.0389 (7)	−0.0008 (7)	−0.0065 (7)	0.0009 (6)
C8	0.0583 (13)	0.0524 (11)	0.0515 (10)	−0.0075 (10)	0.0016 (10)	0.0146 (8)
C9	0.0853 (19)	0.0504 (12)	0.0753 (15)	−0.0096 (13)	−0.0046 (14)	0.0242 (11)
N1	0.0408 (8)	0.0410 (7)	0.0449 (7)	−0.0039 (7)	−0.0071 (7)	0.0092 (6)
N2	0.0407 (8)	0.0291 (6)	0.0414 (7)	0.0036 (6)	−0.0004 (6)	0.0021 (5)
N3	0.0341 (7)	0.0305 (6)	0.0367 (6)	0.0070 (6)	0.0007 (6)	0.0031 (5)
O1	0.0571 (10)	0.0531 (8)	0.0706 (9)	0.0089 (8)	−0.0214 (8)	0.0145 (7)
O2	0.0850 (13)	0.0544 (8)	0.0619 (8)	−0.0040 (9)	−0.0289 (9)	−0.0058 (7)
O3	0.0453 (7)	0.0503 (7)	0.0521 (7)	0.0101 (7)	0.0084 (6)	0.0035 (6)
O4	0.0387 (6)	0.0392 (6)	0.0485 (7)	0.0011 (5)	−0.0035 (6)	0.0120 (5)

Geometric parameters (Å, º)

C1—C2	1.358 (2)	C5—H5A	0.9700
C1—N2	1.360 (2)	C5—H5B	0.9700
C1—N1	1.430 (2)	C6—O3	1.200 (2)
C2—N3	1.361 (2)	C6—O4	1.3194 (19)
C2—H2	0.9300	C8—O4	1.457 (2)
C3—N2	1.317 (2)	C8—C9	1.494 (3)
C3—N3	1.3764 (19)	C8—H8A	0.9700
C3—C4	1.483 (2)	C8—H8B	0.9700
C4—H4A	0.9600	C9—H9A	0.9600
C4—H4B	0.9600	C9—H9B	0.9600
C4—H4C	0.9600	C9—H9C	0.9600
C5—N3	1.455 (2)	N1—O1	1.2200 (19)
C5—C6	1.512 (2)	N1—O2	1.227 (2)
C2—C1—N2	113.02 (14)	O3—C6—C5	124.00 (15)
C2—C1—N1	125.71 (15)	O4—C6—C5	110.36 (14)
N2—C1—N1	121.26 (14)	O4—C8—C9	106.94 (19)
C1—C2—N3	104.13 (14)	O4—C8—H8A	110.3
C1—C2—H2	127.9	C9—C8—H8A	110.3
N3—C2—H2	127.9	O4—C8—H8B	110.3
N2—C3—N3	111.28 (15)	C9—C8—H8B	110.3
N2—C3—C4	125.90 (15)	H8A—C8—H8B	108.6
N3—C3—C4	122.82 (16)	C8—C9—H9A	109.5
C3—C4—H4A	109.5	C8—C9—H9B	109.5
C3—C4—H4B	109.5	H9A—C9—H9B	109.5
H4A—C4—H4B	109.5	C8—C9—H9C	109.5
C3—C4—H4C	109.5	H9A—C9—H9C	109.5
H4A—C4—H4C	109.5	H9B—C9—H9C	109.5
H4B—C4—H4C	109.5	O1—N1—O2	123.66 (16)
N3—C5—C6	111.33 (14)	O1—N1—C1	118.61 (15)
N3—C5—H5A	109.4	O2—N1—C1	117.72 (16)
C6—C5—H5A	109.4	C3—N2—C1	103.94 (13)
N3—C5—H5B	109.4	C2—N3—C3	107.62 (14)
C6—C5—H5B	109.4	C2—N3—C5	124.56 (13)
H5A—C5—H5B	108.0	C3—N3—C5	127.64 (15)
O3—C6—O4	125.64 (17)	C6—O4—C8	115.87 (15)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the imidazole ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O3^{i}	0.97	2.43	3.224 (2)	139
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