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2-Methoxy-1-methyl-4-nitro-1*H*-imidazole

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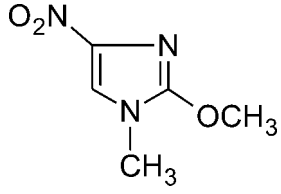
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.001 Å; R factor = 0.033; wR factor = 0.091; data-to-parameter ratio = 14.0.

The molecule of the title compound, $C_5H_7N_3O_3$, is approximately planar. The maximum deviation from the least-squares plane calculated for all non-H atoms is 0.054 (2) Å. The dihedral angles between the mean plane of the imidazole ring [planar within 0.0017 (6) Å] and the planes of the nitro and methoxy groups are 2.9 (1) and 1.2 (1)°, respectively. The molecules are held together by weak C—H···N and C—H···O interactions and by van der Waals forces.

Related literature

This is a part of our studies of intermolecular interactions in 4-nitroimidazole derivatives (*e.g.* Kubicki, 2004, and references therein). Related literature: Kulkarni *et al.* (1987); Suwiński & Wagner (2007).



Experimental

Crystal data

$C_5H_7N_3O_3$
M_r = 157.14
Monoclinic, $P2_1/c$
a = 3.9935 (4) Å
b = 15.4223 (13) Å
c = 11.1724 (8) Å
β = 99.710 (7)°
V = 678.24 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 90 (1) K
0.15 × 0.1 × 0.1 mm

Data collection

Kuma KM-4 CCD four-circle diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1989)
T_{min} = 0.988, T_{max} = 0.991
6537 measured reflections
1791 independent reflections
1514 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
S = 1.08
1791 reflections
128 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O42^{i}	0.967 (15)	2.713 (15)	3.6751 (13)	173.0 (11)
C11—H11C···O41^{ii}	0.947 (17)	2.633 (17)	3.4342 (13)	142.6 (12)
C5—H5···N3^{ii}	0.938 (14)	2.670 (14)	3.5154 (12)	150.2 (11)
C21—H21C···O2^{iii}	0.984 (15)	2.720 (15)	3.6841 (13)	166.5 (11)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2054).

References

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Kulkarni, S., Grimmett, M. R., Hanton, L. R. & Simpson, J. (1987). *Aust. J. Chem.* **40**, 1399–1413.
Oxford Diffraction (2002). *CrysAlis CCD* and *CrysAlis RED*. Version 1.169. Oxford Diffraction, Wrocław, Poland.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
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Suwiński, J. & Wagner, P. (2007). In preparation.

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supplementary materials

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2-Methoxy-1-methyl-4-nitro-1*H*-imidazole

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Comment

The molecule of 1-methyl-2-methoxy-4-nitroimidazole, (**I**), (Fig. 1, Scheme 1) is almost planar, contrary to its analogue 1-methyl-4-nitro-5-methoxyimidazole, (**II**), that was determined at 100 K (Kulkarni *et al.*, 1987; CCDC-258086). In (**II**) the methoxy and and the nitro groups are twisted by 57.55 (14) and by 7.90 (12)°, respectively. These larger twists are obviously concomitant to the steric interactions that have a profound effect on the crystal packing.

In (**I**), there are weak hydrogen bonds that interconnect molecules into layers (Fig. 2). In (**II**), on the other hand, the C—H···A interactions (A = O or N) are somewhat stronger (the H···O distances are in the range 2.38 Å − 2.57 Å, H···N is 2.54 Å. Additionally, a π-π interaction with the interplanar distance of 3.275 (2)Å (the distance between the ring centroids is 3.629 (2) Å) is also present in the structure of (**II**).

Experimental

The title compound was synthesized by *ipso* nucleophilic replacement of nitro group from 2,4-dinitro-1-methyl imidazole in methanol-sodium methoxide solution with a yield (*ca*. 54%). The detailed synthesis will be described elsewhere (Suwiński & Wagner, 2007).

Refinement

All the hydrogen atoms were discernible in the difference Fourier maps and were freely refined.

Figures

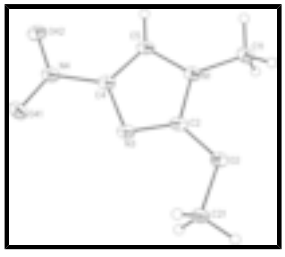

Fig. 1. Title molecule with anisotropic displacement parameters at the 50% probability level. The hydrogen atoms are drawn as spheres with arbitrary radii.

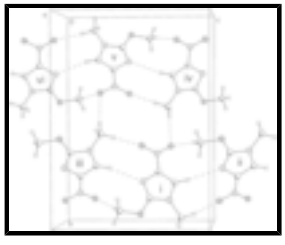

Fig. 2. The layer of the title molecules with C—H···O and C—H···N hydrogen bonds depicted as dashed lines. The view is approximately along the direction [100]. Symmetry codes: (i) *x*,*y*,*z*; (ii): *x*,3/2 − *y*,1/2 + *z*; (iii) *x*,3/2 − *y*,-1/2 + *z*; (iv) 1 − *x*,-1/2 + *y*,3/2 − *z*; (v) 1 − *x*,1 − *y*,1 − *z*; (vi) 1 − *x*,-1/2 + *y*,1/2 − *z*.

2-Methoxy-1-methyl-4-nitro-1*H*-imidazole

Crystal data

$C_5H_7N_3O_3$	F_{000} = 328
M_r = 157.14	D_x = 1.539 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5503 reflections
a = 3.9935 (4) Å	θ = 3–24º
b = 15.4223 (13) Å	μ = 0.13 mm^{-1}
c = 11.1724 (8) Å	T = 90 (1) K
β = 99.710 (7)º	Prism, colourless
V = 678.24 (10) Å^3	0.15 × 0.1 × 0.1 mm
Z = 4	

Data collection

Kuma KM-4 CCD four-circle diffractometer	1791 independent reflections
Radiation source: fine-focus sealed tube	1514 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
T = 90(1) K	θ_{max} = 30.0º
ω scans	θ_{min} = 3.2º
Absorption correction: multi-scan (SORTAV; Blessing, 1989)	h = −5→5
T_{min} = 0.988, T_{max} = 0.991	k = −21→20
6537 measured reflections	l = −7→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.033	All H-atom parameters refined
$wR(F^2)$ = 0.091	$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.0288P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
1791 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
128 parameters	$\Delta\rho_{min}$ = −0.32 e Å^{-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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.9835 (2)	0.86207 (6)	0.74562 (7)	0.01581 (19)
C11	1.0726 (3)	0.93772 (7)	0.82410 (9)	0.0195 (2)
H11A	0.915 (4)	0.9850 (9)	0.8026 (12)	0.028 (3)*
H11B	1.295 (4)	0.9564 (10)	0.8185 (13)	0.034 (4)*
H11C	1.060 (4)	0.9218 (11)	0.9051 (15)	0.037 (4)*
C2	1.0365 (2)	0.85322 (6)	0.62825 (8)	0.0154 (2)
O2	1.19492 (18)	0.91689 (5)	0.57922 (6)	0.01867 (18)
C21	1.2306 (3)	0.90098 (7)	0.45370 (9)	0.0183 (2)
H21A	1.008 (3)	0.8990 (9)	0.4044 (12)	0.024 (3)*
H21B	1.356 (3)	0.8470 (9)	0.4504 (12)	0.021 (3)*
H21C	1.361 (4)	0.9506 (10)	0.4307 (13)	0.029 (3)*
N3	0.9215 (2)	0.78040 (5)	0.57663 (7)	0.01595 (19)
C4	0.7856 (2)	0.74043 (6)	0.66772 (8)	0.0159 (2)
N4	0.6282 (2)	0.65768 (6)	0.64766 (7)	0.01753 (19)
O41	0.6293 (2)	0.62136 (5)	0.54923 (7)	0.0250 (2)
O42	0.49664 (19)	0.62600 (5)	0.73091 (7)	0.02218 (19)
C5	0.8197 (2)	0.78822 (6)	0.77259 (8)	0.0162 (2)
H5	0.760 (3)	0.7775 (9)	0.8489 (12)	0.022 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0179 (4)	0.0188 (4)	0.0110 (4)	0.0008 (3)	0.0032 (3)	−0.0002 (3)
C11	0.0241 (5)	0.0215 (5)	0.0130 (5)	−0.0015 (4)	0.0036 (4)	−0.0034 (4)
C2	0.0159 (4)	0.0199 (5)	0.0109 (4)	0.0023 (3)	0.0033 (3)	0.0017 (3)
O2	0.0245 (4)	0.0205 (4)	0.0122 (3)	−0.0035 (3)	0.0065 (3)	−0.0002 (2)
C21	0.0218 (5)	0.0232 (5)	0.0107 (4)	−0.0008 (4)	0.0050 (3)	0.0006 (3)
N3	0.0180 (4)	0.0185 (4)	0.0116 (4)	0.0011 (3)	0.0034 (3)	0.0013 (3)
C4	0.0171 (4)	0.0187 (4)	0.0122 (4)	0.0009 (3)	0.0031 (3)	0.0011 (3)
N4	0.0199 (4)	0.0205 (4)	0.0125 (4)	0.0007 (3)	0.0035 (3)	0.0004 (3)
O41	0.0360 (4)	0.0250 (4)	0.0150 (4)	−0.0049 (3)	0.0073 (3)	−0.0058 (3)
O42	0.0279 (4)	0.0240 (4)	0.0162 (4)	−0.0036 (3)	0.0082 (3)	0.0031 (3)
C5	0.0180 (4)	0.0192 (5)	0.0117 (4)	0.0009 (3)	0.0035 (3)	0.0013 (3)

Geometric parameters (Å, °)

N1—C2	1.3699 (12)	C21—H21A	0.965 (14)
N1—C5	1.3722 (13)	C21—H21B	0.975 (14)

N1—C11	1.4668 (12)	C21—H21C	0.984 (15)
C11—H11A	0.967 (15)	N3—C4	1.3771 (12)
C11—H11B	0.946 (17)	C4—C5	1.3714 (13)
C11—H11C	0.947 (17)	C4—N4	1.4234 (13)
C2—N3	1.3097 (13)	N4—O41	1.2348 (11)
C2—O2	1.3347 (12)	N4—O42	1.2428 (11)
O2—C21	1.4542 (11)	C5—H5	0.938 (14)
C2—N1—C5	106.49 (8)	H21A—C21—H21B	112.4 (11)
C2—N1—C11	126.19 (8)	O2—C21—H21C	105.2 (8)
C5—N1—C11	127.27 (8)	H21A—C21—H21C	110.7 (12)
N1—C11—H11A	111.6 (8)	H21B—C21—H21C	110.9 (11)
N1—C11—H11B	109.8 (9)	C2—N3—C4	102.19 (8)
H11A—C11—H11B	109.6 (13)	C5—C4—N3	113.33 (9)
N1—C11—H11C	108.6 (10)	C5—C4—N4	126.43 (9)
H11A—C11—H11C	107.2 (13)	N3—C4—N4	120.23 (8)
H11B—C11—H11C	110.0 (13)	O41—N4—O42	123.61 (9)
N3—C2—O2	127.27 (8)	O41—N4—C4	118.63 (8)
N3—C2—N1	114.05 (8)	O42—N4—C4	117.76 (8)
O2—C2—N1	118.68 (8)	C4—C5—N1	103.93 (8)
C2—O2—C21	113.70 (7)	C4—C5—H5	132.7 (9)
O2—C21—H21A	109.0 (8)	N1—C5—H5	123.3 (9)
O2—C21—H21B	108.3 (8)		
C5—N1—C2—N3	0.10 (11)	C2—N3—C4—N4	179.52 (8)
C11—N1—C2—N3	−177.50 (9)	C5—C4—N4—O41	−177.26 (9)
C5—N1—C2—O2	−179.74 (8)	N3—C4—N4—O41	2.98 (13)
C11—N1—C2—O2	2.67 (14)	C5—C4—N4—O42	2.78 (14)
N3—C2—O2—C21	1.33 (13)	N3—C4—N4—O42	−176.99 (8)
N1—C2—O2—C21	−178.86 (8)	N3—C4—C5—N1	0.34 (11)
O2—C2—N3—C4	179.92 (9)	N4—C4—C5—N1	−179.44 (9)
N1—C2—N3—C4	0.10 (10)	C2—N1—C5—C4	−0.25 (10)
C2—N3—C4—C5	−0.28 (11)	C11—N1—C5—C4	177.31 (9)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11A···O42^{i}	0.967 (15)	2.713 (15)	3.6751 (13)	173.0 (11)
C11—H11C···O41^{ii}	0.947 (17)	2.633 (17)	3.4342 (13)	142.6 (12)
C5—H5···N3^{ii}	0.938 (14)	2.670 (14)	3.5154 (12)	150.2 (11)
C21—H21C···O2^{iii}	0.984 (15)	2.720 (15)	3.6841 (13)	166.5 (11)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) −*x*+3, −*y*+2, −*z*+1.

Fig. 1

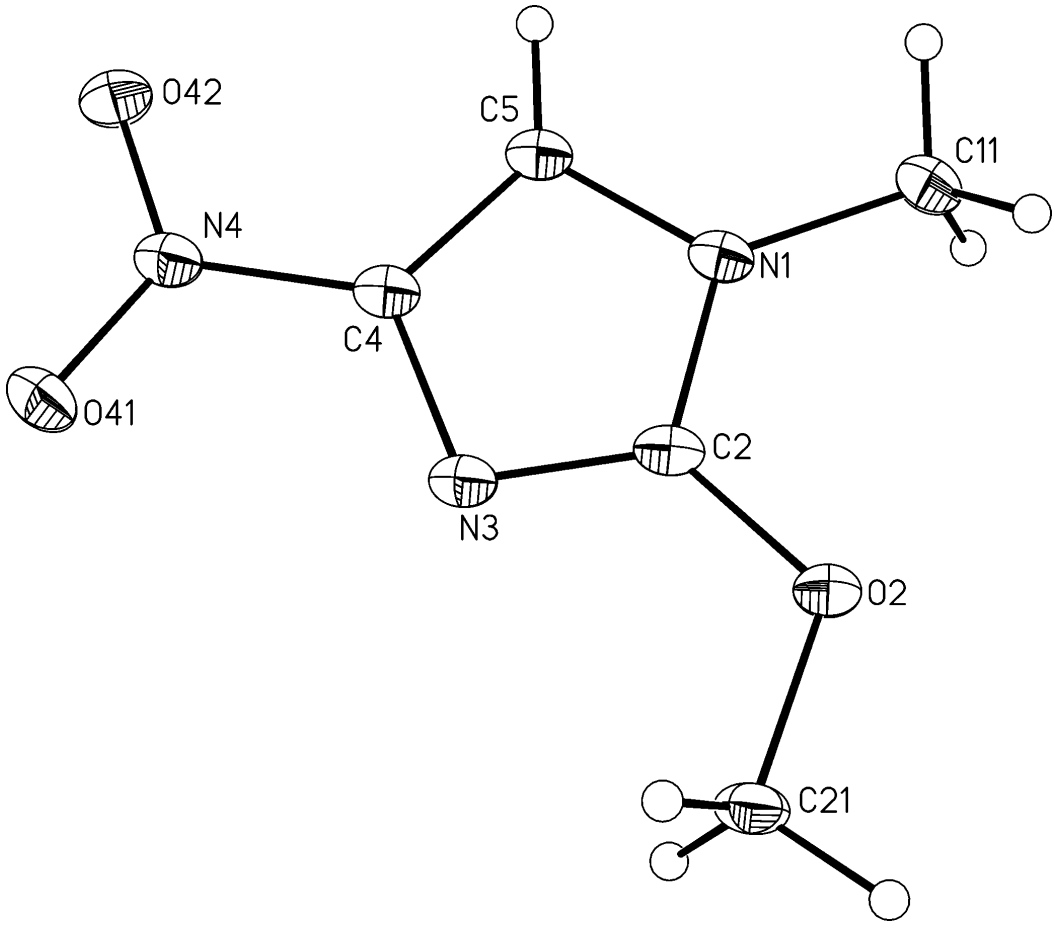

Fig. 2