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

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Abstract
The molecule of the title compound, C$_{5}$H$_{7}$N$_{3}$O$_{3}$, 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.

Keywords
1, 2, 1h, nitro, 4, methyl, imidazole, methoxy

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The molecule of the title compound, C₇H₇N₃O₃, 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. Kulkarni, 2004, and references therein). Related literature: Kulkarni et al. (1987); Suwiński & Wagner (2007).

**Experimental**

**Crystal data**

C₇H₇N₃O₃

Monoclinic, P2₁/c

a = 3.9935 (4) Å

**Table 1**

Hydrogen-bond geometry (Å, °).

<table>
<thead>
<tr>
<th>D—H···A</th>
<th>D—H</th>
<th>H···A</th>
<th>D···A</th>
<th>D—H···A</th>
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</thead>
<tbody>
<tr>
<td>C11—H11A···O42'</td>
<td>0.967 (15)</td>
<td>2.713 (15)</td>
<td>3.6751 (13)</td>
<td>173.0 (11)</td>
</tr>
<tr>
<td>C11—H11C···O41'</td>
<td>0.947 (17)</td>
<td>2.633 (17)</td>
<td>3.4342 (13)</td>
<td>142.6 (12)</td>
</tr>
<tr>
<td>C5—H5···N3'</td>
<td>0.938 (14)</td>
<td>2.670 (14)</td>
<td>3.5154 (12)</td>
<td>150.2 (11)</td>
</tr>
<tr>
<td>C21—H21C···O22'</td>
<td>0.984 (15)</td>
<td>2.720 (15)</td>
<td>3.6843 (13)</td>
<td>166.5 (11)</td>
</tr>
</tbody>
</table>

Symmetry codes: (i) x + 1, y + ½, z; (ii) x, y + ½, z; (iii) x + 3, y + 2, z + 1.

Data collection: CrysAlis CCD (Oxford Diffraction, 2002); cell refinement: 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**


supplementary materials
2-Methoxy-1-methyl-4-nitro-1H-imidazole

M. Kubicki and P. Wagner

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 − y,1 − z; (v) 1 − x,1 − y,1 − z; (vi) 1 − x,−1/2 + y,1/2 − z.
2-Methoxy-1-methyl-4-nitro-1H-imidazole

Crystal data

\( \text{C}_{5}\text{H}_{7}\text{N}_{3}\text{O}_{3} \)

\( F_{000} = 328 \)

\( M_r = 157.14 \)

\( D_x = 1.539 \text{ Mg m}^{-3} \)

Monoclinic, \( P2_1/c \)

Hall symbol: -P 2\( y \)bc

Cell parameters from 5503 reflections

\( a = 3.9935 (4) \text{ Å} \)

\( 0 = 3–24^\circ \)

\( b = 15.4223 (13) \text{ Å} \)

\( \mu = 0.13 \text{ mm}^{-1} \)

\( c = 11.1724 (8) \text{ Å} \)

\( T = 90 (1) \text{ K} \)

\( \beta = 99.710 (7)^\circ \)

Prism, colourless

\( V = 678.24 (10) \text{ Å}^3 \)

\( 0.15 \times 0.1 \times 0.1 \text{ mm} \)

\( Z = 4 \)

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

\( R_{int} = 0.019 \)

\( \theta_{max} = 30.0^\circ \)

\( 0 = 3–24^\circ \)

\( \omega \) scans

Absorption correction: multi-scan

SORTAV; Blessing, 1989

\( h = -5 \rightarrow 5 \)

\( k = -21 \rightarrow 20 \)

\( l = -7 \rightarrow 15 \)

6537 measured reflections

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

\( wR(F^2) = 0.091 \)

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

\( (\Delta\sigma)_{max} = 0.001 \)

1791 reflections

\( \Delta\rho_{max} = 0.25 \text{ e Å}^{-3} \)

128 parameters

\( \Delta\rho_{min} = -0.32 \text{ e Å}^{-3} \)

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

sup-2
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 F² against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F², conventional R-factors R are based on F, with F set to zero for negative F². The threshold expression of F² > 2σ(F²) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F² 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 (Å²)

<table>
<thead>
<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Uiso* or Ueq</th>
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<td>0.9835 (2)</td>
<td>0.86207 (6)</td>
<td>0.74562 (7)</td>
<td>0.01581 (19)</td>
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<tr>
<td>C11</td>
<td>1.0726 (3)</td>
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<td>0.82410 (9)</td>
<td>0.0195 (2)</td>
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<tr>
<td>H11A</td>
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<td>0.9850 (9)</td>
<td>0.8026 (12)</td>
<td>0.028 (3)*</td>
</tr>
<tr>
<td>H11B</td>
<td>1.295 (4)</td>
<td>0.9564 (10)</td>
<td>0.8185 (13)</td>
<td>0.034 (4)*</td>
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<tr>
<td>H11C</td>
<td>1.060 (4)</td>
<td>0.9218 (11)</td>
<td>0.9051 (15)</td>
<td>0.037 (4)*</td>
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<tr>
<td>C2</td>
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<td>H21A</td>
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<tr>
<td>H21B</td>
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<td>0.8470 (9)</td>
<td>0.4504 (12)</td>
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<td>N3</td>
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<td>0.78040 (5)</td>
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<td>C4</td>
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<td>0.74043 (6)</td>
<td>0.66772 (8)</td>
<td>0.0159 (2)</td>
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<td>N4</td>
<td>0.6282 (2)</td>
<td>0.65768 (6)</td>
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<td>O41</td>
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<td>O42</td>
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<td>0.7775 (9)</td>
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<td>0.022 (3)*</td>
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Atomic displacement parameters (Å²)

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<thead>
<tr>
<th>Atom</th>
<th>U₁₁</th>
<th>U₂₂</th>
<th>U₃₃</th>
<th>U₁₂</th>
<th>U₁₃</th>
<th>U₂₃</th>
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<tr>
<td>N1</td>
<td>0.0179 (4)</td>
<td>0.0188 (4)</td>
<td>0.0110 (4)</td>
<td>0.0008 (3)</td>
<td>0.0032 (3)</td>
<td>0.0002 (3)</td>
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<tr>
<td>C11</td>
<td>0.0241 (5)</td>
<td>0.0215 (5)</td>
<td>0.0130 (5)</td>
<td>−0.0015 (4)</td>
<td>0.0036 (4)</td>
<td>−0.0034 (4)</td>
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<td>C2</td>
<td>0.0159 (4)</td>
<td>0.0199 (5)</td>
<td>0.0109 (4)</td>
<td>0.0023 (3)</td>
<td>0.0033 (3)</td>
<td>0.0017 (3)</td>
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<tr>
<td>O2</td>
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<td>0.0205 (4)</td>
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<td>0.0065 (3)</td>
<td>−0.0002 (2)</td>
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<tr>
<td>C21</td>
<td>0.0218 (5)</td>
<td>0.0232 (5)</td>
<td>0.0107 (4)</td>
<td>−0.0008 (4)</td>
<td>0.0050 (3)</td>
<td>0.0006 (3)</td>
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<td>N3</td>
<td>0.0180 (4)</td>
<td>0.0185 (4)</td>
<td>0.0116 (4)</td>
<td>0.0011 (3)</td>
<td>0.0034 (3)</td>
<td>0.0013 (3)</td>
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<td>0.0171 (4)</td>
<td>0.0187 (4)</td>
<td>0.0122 (4)</td>
<td>0.0009 (3)</td>
<td>0.0031 (3)</td>
<td>0.0011 (3)</td>
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<td>N4</td>
<td>0.0199 (4)</td>
<td>0.0205 (4)</td>
<td>0.0125 (4)</td>
<td>0.0007 (3)</td>
<td>0.0035 (3)</td>
<td>0.0004 (3)</td>
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<td>0.0150 (4)</td>
<td>−0.0049 (3)</td>
<td>0.0073 (3)</td>
<td>−0.0058 (3)</td>
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<tr>
<td>O42</td>
<td>0.0279 (4)</td>
<td>0.0240 (4)</td>
<td>0.0162 (4)</td>
<td>−0.0036 (3)</td>
<td>0.0082 (3)</td>
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<tr>
<td>C5</td>
<td>0.0180 (4)</td>
<td>0.0192 (5)</td>
<td>0.0117 (4)</td>
<td>0.0009 (3)</td>
<td>0.0035 (3)</td>
<td>0.0013 (3)</td>
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</table>

Geometric parameters (Å, °)

<table>
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<tr>
<th>Bond</th>
<th>Distance (Å)</th>
<th>Angle (°)</th>
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<tbody>
<tr>
<td>N1—C2</td>
<td>1.3699 (12)</td>
<td>C21—H21A</td>
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<tr>
<td>N1—C5</td>
<td>1.3722 (13)</td>
<td>C21—H21B</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.965 (14)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.975 (14)</td>
</tr>
</tbody>
</table>
supplementary materials

N1—C11 1.4668 (12)  C21—H21C 0.984 (15)  N984 (15)
C11—H11A 0.967 (15)  N3—C4  1.3771 (12)  1.3714 (13)
C11—H11B 0.946 (17)  C4—C5  1.3714 (13)  1.2348 (11)
C11—H11C 0.947 (17)  C4—N4  1.4234 (13)  1.2428 (11)
C2—N3  1.3097 (13)  N4—O41 1.2348 (11)  0.938 (14)
C2—O2  1.3347 (12)  N4—O42 1.2428 (11)  C5—H5  1.4542 (11)
N1—C11—H11A 111.6 (8) H21A—C21—H21C 110.7 (12) 110.9 (11)
N1—C11—H11B 109.8 (9) C2—N3—C4 102.19 (8)
C5—N1—C11 127.27 (8) C5—C4—N3 113.33 (9) 0.10 (11)
C11—N1—C2—N3 0.10 (11) C5—N1—C2—O2 118.68 (8) 177.27 (8)
C11—N1—C2—O2 179.74 (8) C5—N1—C2—N3 179.92 (9) 0.10 (10)
C5—N1—C2—N3 179.50 (9) C11—N1—C2—N3 179.52 (8) 179.26 (9)
C5—N1—C2—O2 179.92 (9) C11—N1—C2—O2 179.92 (9) 0.34 (11)
C5—N1—C2—N3 179.92 (9) C11—N1—C2—N3 179.92 (9) −179.44 (9)
C5—N1—C2—O2 179.92 (9) C5—N1—C2—N3 179.92 (9) −0.25 (10)
C5—N1—C2—N3 179.92 (9) C5—N1—C2—O2 179.92 (9) −177.31 (9)

Hydrogen-bond geometry (Å, °)

|  | D—H—A         | D—H  | H—A  | D···A        | D—H—A |
|  |   | (Å) | (Å)  | (Å)         | (Å)   |
|  | C11—H11A···O42i 0.967 (15) | 2.713 (15) | 3.6751 (13) | 173.0 (11) |
|  | C11—H11C···O41ii 0.947 (17) | 2.633 (17) | 3.4342 (13) | 142.6 (12) |
|  | C5—H5···N3ii 0.938 (14) | 2.670 (14) | 3.5154 (12) | 150.2 (11) |
|  | C21—H21C···O2iii 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.