

University of Wollongong

Research Online

Australian Institute for Innovative Materials -
Papers

Australian Institute for Innovative Materials

2007

2-Methyl-4-nitro-1-(4-nitrophenyl)-1H-imidazole

Pawel Wagner

University of Wollongong, pawel@uow.edu.au

Maciej Kubicki

Adam Mickiewicz University

Follow this and additional works at: <https://ro.uow.edu.au/aiimpapers>



Part of the [Engineering Commons](#), and the [Physical Sciences and Mathematics Commons](#)

Research Online is the open access institutional repository for the University of Wollongong. For further information contact the UOW Library: research-pubs@uow.edu.au

2-Methyl-4-nitro-1-(4-nitrophenyl)-1H-imidazole

Abstract

In the title compound, C₁₀H₈N₄O₄, two planar fragments, *viz.* the imidazole and nitrophenyl rings, are tilted at a dihedral of 57.89 (7)°. The nitro groups are twisted with respect to the neighbouring ring planes; the dihedral angle is 7.0 (3)° for imidazole and 9.68 (8)° for benzene. The crystal structure consists of centrosymmetric dimers generated by C–H...O hydrogen bonds, which are connected by C–H...N hydrogen bonds into rows along the [001] direction. The neighbouring rows are connected *via* C–H...O interactions into a two-dimensional network in the *bc* plane.

Keywords

nitro, 1, 4, nitrophenyl, 2, 1h, imidazole, methyl

Disciplines

Engineering | Physical Sciences and Mathematics

Publication Details

Wagner, P. & Kubicki, M. (2007). 2-Methyl-4-nitro-1-(4-nitrophenyl)-1H-imidazole. *Acta Crystallographica Section E: Structure Reports Online*, 63 (7), o3083.

2-Methyl-4-nitro-1-(4-nitrophenyl)-1H-imidazole

Paweł Wagner^a and Maciej Kubicki^{b*}

^aNanomaterials Research Centre, and MacDiarmid Institute for Advanced Materials and Nanotechnology, Massey University, Private Bag 11 222, Palmerston North, New Zealand, and ^bDepartment of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland

Correspondence e-mail: mkubicki@amu.edu.pl

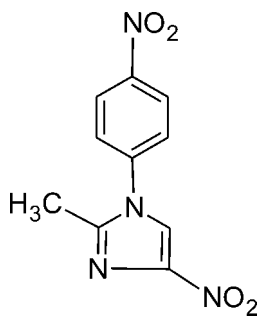
Received 18 May 2007; accepted 29 May 2007

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.038; wR factor = 0.099; data-to-parameter ratio = 12.8.

In the title compound, $\text{C}_{10}\text{H}_8\text{N}_4\text{O}_4$, two planar fragments, *viz.* the imidazole and nitrophenyl rings, are tilted at a dihedral of $57.89(7)^\circ$. The nitro groups are twisted with respect to the neighbouring ring planes; the dihedral angle is $7.0(3)^\circ$ for imidazole and $9.68(8)^\circ$ for benzene. The crystal structure consists of centrosymmetric dimers generated by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, which are connected by $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds into rows along the $[001]$ direction. The neighbouring rows are connected *via* $\text{C}-\text{H}\cdots\text{O}$ interactions into a two-dimensional network in the bc plane.

Related literature

This is a part of our studies of intermolecular interactions on 4-nitroimidazole derivatives (Kubicki, 2004). The crystal structures of other 1-aryl-2-methyl-4-nitroimidazoles were described by Kowalski (1995, 1996), Kubicki (2004) and Kubicki & Wagner (2007). For related literature, see: Suwiński *et al.* (1993).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{N}_4\text{O}_4$	$V = 2150.6(3) \text{ \AA}^3$
$M_r = 248.20$	$Z = 8$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 8.1416(9) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$b = 12.7121(10) \text{ \AA}$	$T = 100(1) \text{ K}$
$c = 20.7789(13) \text{ \AA}$	$0.4 \times 0.15 \times 0.05 \text{ mm}$

Data collection

Kuma KM-4-CCD four-circle diffractometer	11596 measured reflections
Absorption correction: multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2007)	2092 independent reflections
$T_{\min} = 0.918$, $T_{\max} = 1.000$	1442 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.054$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	163 parameters
$wR(F^2) = 0.099$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
2092 reflections	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}12-\text{H}12\cdots\text{N}3^{\text{i}}$	0.94	2.40	3.338 (2)	177
$\text{C}15-\text{H}15\cdots\text{O}42^{\text{ii}}$	0.91	2.55	3.333 (2)	145
$\text{C}21-\text{H}21\text{A}\cdots\text{O}141^{\text{iii}}$	0.97	2.52	3.429 (2)	156
$\text{C}5-\text{H}5\cdots\text{O}141^{\text{iv}}$	0.93	2.46	3.258 (2)	143

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; 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: KP2111).

References

- Kowalski, A. (1995). *Acta Cryst.* **C51**, 1670–1672.
 Kowalski, A. (1996). *Acta Cryst.* **C52**, 1784–1787.
 Kubicki, M. (2004). *Acta Cryst.* **C60**, o341–o343.
 Kubicki, M. & Wagner, P. (2007). *Acta Cryst.* **E63**. In the press.
 Oxford Diffraction (2007). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.171.31.8. Oxford Diffraction, Wrocław, Poland.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Siemens (1989). *Stereochemical Workstation Operation Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Suwiński, J., Świerczek, K. & Głowiak, T. (1993). *Tetrahedron*, **49**, 5339–5350.

supplementary materials

Acta Cryst. (2007). E63, o3083 [doi:10.1107/S1600536807026244]

2-Methyl-4-nitro-1-(4-nitrophenyl)-1*H*-imidazole

P. Wagner and M. Kubicki

Comment

Simple 4-nitroimidzoles are convenient compounds for studying weak interactions in molecular crystals. The molecules are relatively simple, with few degrees of conformational freedom, and the appropriate choice of substituents can highlight certain kind of interaction. Here, in 1-(4-nitrophenyl)-2-methyl-4-nitroimidazole the available specific interactions range from π - π interactions, through weak hydrogen bonds to nitro-nitro interactions. It turned out that the crystal packing is determined by relatively short and directional C—H \cdots N and C—H \cdots O hydrogen bonds. The first level of organization is the centrosymmetric C—H \cdots O bonded dimer. These dimers are connected by C—H \cdots N hydrogen bonds along [001] direction producing the tapes which are connected into two-dimensional network in the *bc* plane. The plane of imidazole ring makes the angle of 57.89° with the benzene ring.

Experimental

The title compound has been synthesized by aromatic nucleophilic substitution by 4(5)-nitro-2-methylimidazole to 4-fluoronitrobenzene catalysed by sodium hydroxide [Suwiński *et al.*, (1993)].

Refinement

Hydrogen atoms were freely refined, and then, for the last cycles of refinement, both the refined geometry and displacement parameters were restrained to keep the refined values.

Figures

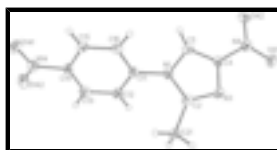


Fig. 1. Molecular structure of (I) with displacement parameters scaled at the 50% probability level (Siemens, 1989) and numbering scheme. The hydrogen atoms are drawn as spheres of arbitrary radii.



Fig. 2. The two-dimensional hydrogen-bonded network of (I) along *a* axis. Hydrogen bonds are drawn as dashed lines.

2-Methyl-4-nitro-1-(4-nitrophenyl)-1*H*-imidazole

Crystal data

C₁₀H₈N₄O₄

M_r = 248.20

*F*₀₀₀ = 1024

D_x = 1.533 Mg m⁻³

supplementary materials

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 8.1416$ (9) Å

$b = 12.7121$ (10) Å

$c = 20.7789$ (13) Å

$V = 2150.6$ (3) Å³

$Z = 8$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3523 reflections

$\theta = 4\text{--}22^\circ$

$\mu = 0.12$ mm⁻¹

$T = 100$ (1) K

Needle, colourless

$0.4 \times 0.15 \times 0.05$ mm

Data collection

Kuma KM-4-CCD four-circle diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100$ (1) K

ω scan

Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)

$T_{\min} = 0.918$, $T_{\max} = 1.000$

11596 measured reflections

2092 independent reflections

1442 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.054$

$\theta_{\max} = 26.0^\circ$

$\theta_{\min} = 3.2^\circ$

$h = -9 \rightarrow 10$

$k = -15 \rightarrow 14$

$l = -25 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.099$

$S = 1.01$

2092 reflections

163 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

$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.1988P]$

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

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

$\Delta\rho_{\max} = 0.23$ e Å⁻³

$\Delta\rho_{\min} = -0.24$ e Å⁻³

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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.88142 (17)	0.07302 (11)	0.63572 (7)	0.0169 (4)
C11	0.7321 (2)	0.09541 (14)	0.60164 (8)	0.0157 (4)
C12	0.5883 (2)	0.11064 (14)	0.63618 (9)	0.0179 (4)
H12	0.5884	0.1074	0.6814	0.022*
C13	0.4431 (2)	0.13081 (13)	0.60316 (9)	0.0190 (4)
H13	0.3489	0.1428	0.6262	0.013*
C14	0.4484 (2)	0.13426 (13)	0.53655 (9)	0.0166 (4)
N14	0.29294 (19)	0.14829 (11)	0.50133 (8)	0.0205 (4)
O141	0.29927 (17)	0.16023 (10)	0.44276 (6)	0.0265 (3)
O142	0.16320 (16)	0.14491 (10)	0.53195 (7)	0.0287 (4)
C15	0.5904 (2)	0.12051 (13)	0.50162 (9)	0.0171 (4)
H15	0.5864	0.1234	0.4579	0.018*
C16	0.7355 (2)	0.10102 (14)	0.53454 (8)	0.0176 (4)
H16	0.8411	0.0954	0.5120	0.025*
C2	0.9542 (2)	0.13460 (13)	0.68233 (9)	0.0172 (4)
C21	0.8855 (2)	0.23579 (14)	0.70574 (9)	0.0204 (4)
H21A	0.8362	0.2761	0.6711	0.051*
H21B	0.9711	0.2734	0.7232	0.036*
H21C	0.8029	0.2243	0.7389	0.028*
N3	1.09046 (17)	0.09022 (12)	0.70367 (7)	0.0174 (3)
C4	1.1017 (2)	-0.00159 (14)	0.66976 (8)	0.0160 (4)
N4	1.23301 (18)	-0.07395 (12)	0.68067 (7)	0.0200 (4)
O41	1.32853 (17)	-0.05715 (11)	0.72537 (6)	0.0293 (4)
O42	1.24362 (16)	-0.15076 (10)	0.64383 (6)	0.0258 (3)
C5	0.9775 (2)	-0.01455 (14)	0.62716 (8)	0.0184 (4)
H5	0.9442	-0.0686	0.5998	0.019*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0150 (8)	0.0190 (8)	0.0167 (8)	-0.0009 (6)	0.0001 (6)	0.0012 (6)
C11	0.0159 (9)	0.0134 (9)	0.0177 (9)	-0.0014 (7)	-0.0023 (8)	0.0006 (8)
C12	0.0179 (9)	0.0220 (10)	0.0138 (9)	-0.0015 (8)	-0.0004 (8)	0.0009 (8)
C13	0.0169 (10)	0.0185 (10)	0.0216 (10)	-0.0025 (7)	0.0054 (8)	-0.0007 (8)
C14	0.0145 (9)	0.0134 (9)	0.0219 (10)	-0.0012 (7)	-0.0063 (8)	0.0000 (8)
N14	0.0214 (9)	0.0147 (8)	0.0255 (9)	-0.0013 (6)	-0.0036 (7)	0.0008 (7)
O141	0.0332 (9)	0.0256 (8)	0.0207 (7)	-0.0006 (6)	-0.0100 (6)	0.0030 (6)
O142	0.0209 (7)	0.0293 (8)	0.0360 (9)	-0.0005 (6)	-0.0024 (7)	0.0065 (7)
C15	0.0224 (10)	0.0146 (9)	0.0143 (9)	-0.0033 (8)	-0.0016 (8)	0.0010 (7)
C16	0.0187 (9)	0.0158 (10)	0.0181 (10)	-0.0018 (7)	0.0020 (8)	0.0000 (8)
C2	0.0191 (10)	0.0174 (9)	0.0151 (9)	-0.0046 (7)	0.0016 (8)	0.0006 (8)
C21	0.0188 (10)	0.0218 (10)	0.0206 (10)	-0.0026 (8)	-0.0009 (8)	-0.0025 (8)
N3	0.0190 (8)	0.0193 (8)	0.0141 (8)	-0.0037 (7)	-0.0001 (6)	0.0007 (6)
C4	0.0144 (9)	0.0171 (9)	0.0165 (10)	-0.0007 (7)	0.0015 (8)	0.0017 (7)

supplementary materials

N4	0.0186 (8)	0.0223 (9)	0.0192 (8)	-0.0031 (7)	0.0000 (7)	0.0019 (7)
O41	0.0266 (8)	0.0300 (8)	0.0312 (8)	0.0016 (6)	-0.0140 (7)	-0.0031 (6)
O42	0.0278 (8)	0.0229 (7)	0.0266 (8)	0.0022 (6)	0.0009 (6)	-0.0056 (6)
C5	0.0232 (10)	0.0157 (9)	0.0162 (9)	-0.0005 (8)	0.0018 (8)	-0.0008 (8)

Geometric parameters (Å, °)

N1—C5	1.372 (2)	C15—H15	0.9093
N1—C2	1.379 (2)	C16—H16	0.9824
N1—C11	1.436 (2)	C2—N3	1.321 (2)
C11—C12	1.387 (2)	C2—C21	1.485 (2)
C11—C16	1.396 (2)	C21—H21A	0.9710
C12—C13	1.390 (2)	C21—H21B	0.9201
C12—H12	0.9414	C21—H21C	0.9739
C13—C14	1.386 (2)	N3—C4	1.366 (2)
C13—H13	0.9168	C4—C5	1.354 (2)
C14—C15	1.376 (2)	C4—N4	1.428 (2)
C14—N14	1.473 (2)	N4—O41	1.2301 (19)
N14—O141	1.2274 (19)	N4—O42	1.2436 (19)
N14—O142	1.2339 (19)	C5—H5	0.9323
C15—C16	1.387 (2)		
C5—N1—C2	107.85 (15)	C15—C16—H16	121.6
C5—N1—C11	125.42 (15)	C11—C16—H16	119.3
C2—N1—C11	126.72 (15)	N3—C2—N1	110.74 (15)
C12—C11—C16	121.75 (16)	N3—C2—C21	125.22 (16)
C12—C11—N1	119.16 (15)	N1—C2—C21	124.02 (16)
C16—C11—N1	119.08 (15)	C2—C21—H21A	111.8
C11—C12—C13	119.20 (17)	C2—C21—H21B	107.1
C11—C12—H12	120.7	H21A—C21—H21B	109.3
C13—C12—H12	120.1	C2—C21—H21C	111.3
C14—C13—C12	118.21 (16)	H21A—C21—H21C	108.6
C14—C13—H13	122.8	H21B—C21—H21C	108.7
C12—C13—H13	119.0	C2—N3—C4	104.37 (15)
C15—C14—C13	123.28 (16)	C5—C4—N3	113.01 (16)
C15—C14—N14	118.37 (16)	C5—C4—N4	125.75 (16)
C13—C14—N14	118.28 (16)	N3—C4—N4	121.24 (15)
O141—N14—O142	123.47 (16)	O41—N4—O42	123.86 (16)
O141—N14—C14	118.14 (15)	O41—N4—C4	118.76 (15)
O142—N14—C14	118.37 (16)	O42—N4—C4	117.38 (15)
C14—C15—C16	118.56 (17)	C4—C5—N1	104.02 (15)
C14—C15—H15	119.4	C4—C5—H5	134.9
C16—C15—H15	122.0	N1—C5—H5	120.8
C15—C16—C11	118.99 (16)		
C5—N1—C11—C12	122.19 (19)	N1—C11—C16—C15	178.68 (15)
C2—N1—C11—C12	-58.0 (2)	C5—N1—C2—N3	-0.3 (2)
C5—N1—C11—C16	-57.6 (2)	C11—N1—C2—N3	179.90 (15)
C2—N1—C11—C16	122.18 (19)	C5—N1—C2—C21	-179.11 (16)
C16—C11—C12—C13	0.8 (3)	C11—N1—C2—C21	1.0 (3)
N1—C11—C12—C13	-179.07 (15)	N1—C2—N3—C4	-0.44 (19)

C11—C12—C13—C14	0.4 (2)	C21—C2—N3—C4	178.40 (17)
C12—C13—C14—C15	-1.1 (3)	C2—N3—C4—C5	1.0 (2)
C12—C13—C14—N14	175.83 (15)	C2—N3—C4—N4	-178.39 (15)
C15—C14—N14—O141	-8.9 (2)	C5—C4—N4—O41	-172.79 (17)
C13—C14—N14—O141	173.95 (15)	N3—C4—N4—O41	6.5 (2)
C15—C14—N14—O142	169.37 (15)	C5—C4—N4—O42	7.3 (3)
C13—C14—N14—O142	-7.8 (2)	N3—C4—N4—O42	-173.35 (15)
C13—C14—C15—C16	0.8 (3)	N3—C4—C5—N1	-1.2 (2)
N14—C14—C15—C16	-176.21 (15)	N4—C4—C5—N1	178.22 (15)
C14—C15—C16—C11	0.4 (2)	C2—N1—C5—C4	0.84 (18)
C12—C11—C16—C15	-1.1 (3)	C11—N1—C5—C4	-179.31 (15)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C12—H12 \cdots N3 ⁱ	0.94	2.40	3.338 (2)	177
C15—H15 \cdots O42 ⁱⁱ	0.91	2.55	3.333 (2)	145
C21—H21A \cdots O141 ⁱⁱⁱ	0.97	2.52	3.429 (2)	156
C5—H5 \cdots O141 ^{iv}	0.93	2.46	3.258 (2)	143

Symmetry codes: (i) $x-1/2, y, -z+3/2$; (ii) $-x+2, -y, -z+1$; (iii) $x+1/2, -y+1/2, -z+1$; (iv) $-x+1, -y, -z+1$.

Fig. 1

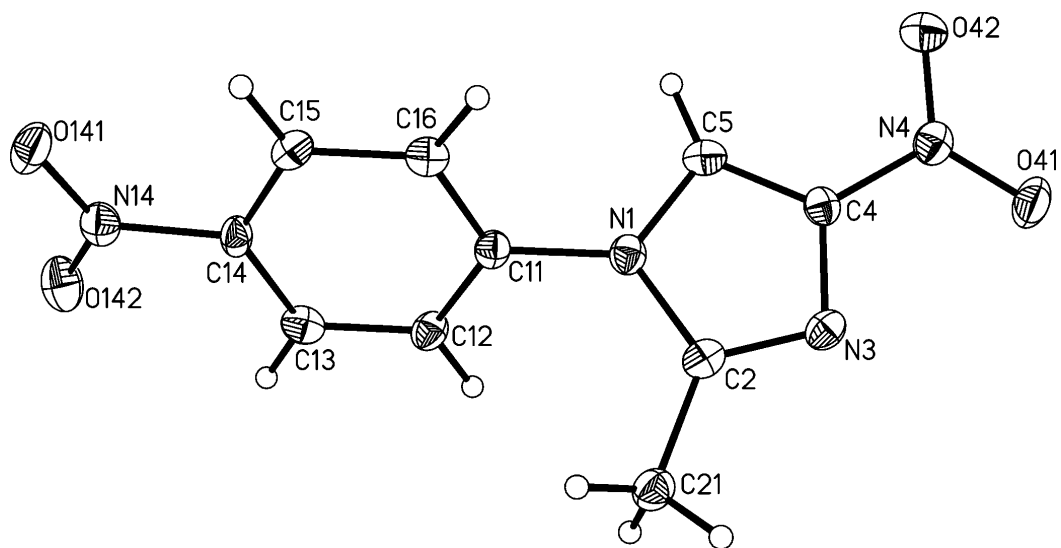


Fig. 2

