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2-Methyl-4-nitro-1-(3-pyridyl)-1H-imidazole

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2-Methyl-4-nitro-1-(3-pyridyl)-1H-imidazole

Abstract

The imidazole and pyridine rings in the title compound, $C_9H_8N_4O_4$, are twisted with respect to one another, with a dihedral angle of 48.30 (4)°. The nitro group is almost coplanar with the imidazole plane. The crystal packing involves some weak C—H...N and C—H...O hydrogen bonds, of which the strongest, between the imidazole CH group and a nitro O atom [H...O 2.396 (15) Å], forms a centrosymmetric dimer.

Keywords

1, 3, pyridyl, 1h, imidazole, 4, 2, nitro, methyl

Disciplines

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2-Methyl-4-nitro-1-(3-pyridyl)-1*H*-imidazole

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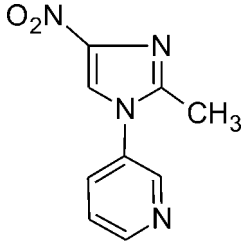
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.123; data-to-parameter ratio = 16.6.

The imidazole and pyridine rings in the title compound, $C_9H_8N_4O_4$, are twisted with respect to one another, with a dihedral angle of 48.30 (4)°. The nitro group is almost coplanar with the imidazole plane. The crystal packing involves some weak C—H···N and C—H···O hydrogen bonds, of which the strongest, between the imidazole CH group and a nitro O atom [H···O 2.396 (15) Å], forms a centrosymmetric dimer.

Related literature

This is a part of our studies of intermolecular interactions in 4-nitroimidazole derivatives that started with 1-phenyl-4-nitroimidazole (Kubicki *et al.*, 2001, 2002). Similar packing schemes and unit-cell parameters were found for 1-phenyl- and 1-(*p*-methylphenyl)-2-methyl-4-nitroimidazole (Kowalski, 1995). For related literature, see: Suwiński & Szczepankiewicz (1991).



Experimental

Crystal data

$C_9H_8N_4O_2$
M_r = 204.19
Monoclinic, $P2_1/n$
a = 8.1315 (12) Å
b = 7.3189 (10) Å
c = 15.104 (3) Å
β = 98.178 (14)°
V = 889.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 291 (1) K
0.5 × 0.4 × 0.1 mm

Data collection

Kuma KM4 CCD four-circle diffractometer
Absorption correction: none
5973 measured reflections
2311 independent reflections
1892 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.123
S = 1.06
2311 reflections
139 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O41^{i}	0.93	2.57	3.2872 (16)	135
C14—H14···N13^{ii}	0.93	2.75	3.5557 (17)	145
C15—H15···N3^{iii}	0.93	2.58	3.4342 (16)	153
C5—H5···O42^{iv}	0.93	2.37	3.2358 (16)	155

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

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: RK2016).

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supplementary materials

Acta Cryst. (2007). E**63**, o3454 [**doi:10.1107/S1600536807023884**]

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Comment

Almost identical crystal packing was observed in the crystal structure of 1-(4-methylphenyl)-2-methyl-4-nitroimidazole (Kowalski, 1995), which crystallizes in similar unit cell (8.259 (2) Å, 7.805 (2) Å, 16.774 (3) Å). Also, there are close analogies between the intermolecular contacts in these structures. Another similar unit cell was used to describe 1-phenyl-2-methyl-4-nitroimidazole (Kowalski, 1995). In this case, however, only the projection along b-direction might be compared to the former cases; the packing along other directions looks quite different. In this case there is less intermolecular contacts in the crystal structure. This comparison might be regarded as another argument in favour of the role played by C—H···O and C—H···N hydrogen bonds in the determination of the crystal packing.

Experimental

The title compound was synthesized by aromatic nucleophilic sybstitution *ANRORC* according to procedure described before (Suwiński & Szczepankiewicz, 1991).

Refinement

Isotropic displacement parameters for hydrogen atoms were calculeted as 1.2 (1.4 for the methyl group) times the U_{eq} value of the appropriate carrier atom.

Figures

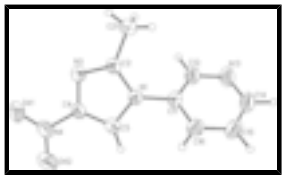

Fig. 1. Anisotropic displacement ellipsoid representation (at the 50% probability level) of the molecule 1 (Siemens, 1989), together with numbering scheme. The hydrogen atoms are drawn as spheres with arbitrary radii.

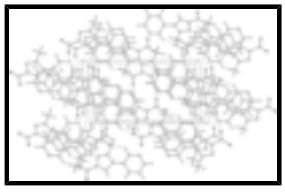

Fig. 2. The crystal packing as seen along [010] direction. Weak hydrogen bonds are depicted as dashed lines.

2-Methyl-4-nitro-1-(3-pyridyl)-1*H*-imidazole

Crystal data

$C_9H_8N_4O_2$ $F_{000} = 424$

M_r = 204.19	D_x = 1.524 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3725 reflections
a = 8.1315 (12) Å	θ = 3–23º
b = 7.3189 (10) Å	μ = 0.11 mm^{-1}
c = 15.104 (3) Å	T = 291 (1) K
β = 98.178 (14)º	Prism, colourless
V = 889.8 (3) Å^3	0.5 × 0.4 × 0.1 mm
Z = 4	

Data collection

KUMA KM4CCD four-circle diffractometer	1892 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.018
Monochromator: graphite	θ_{max} = 29.6º
T = 291(1) K	θ_{min} = 5.1º
ω scan	h = −9→11
Absorption correction: none	k = −10→9
5973 measured reflections	l = −19→20
2311 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.041	$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.1177P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.123	$(\Delta/\sigma)_{max} < 0.001$
S = 1.06	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
2311 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
139 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.049 (8)
Secondary atom site location: difference Fourier map	

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 calculat-

ing *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	0.63363 (11)	0.17729 (13)	0.85813 (6)	0.0313 (2)
C11	0.64183 (13)	0.15842 (14)	0.76563 (7)	0.0312 (2)
C12	0.50520 (15)	0.10274 (17)	0.70904 (8)	0.0397 (3)
H12	0.4073	0.0812	0.7325	0.048*
N13	0.50567 (14)	0.07805 (17)	0.62254 (7)	0.0483 (3)
C14	0.64606 (18)	0.10848 (19)	0.59085 (8)	0.0476 (3)
H14	0.6477	0.0919	0.5299	0.057*
C15	0.78857 (17)	0.16272 (19)	0.64222 (9)	0.0453 (3)
H15	0.8850	0.1821	0.6170	0.054*
C16	0.78730 (14)	0.18828 (17)	0.73201 (8)	0.0387 (3)
H16	0.8827	0.2249	0.7691	0.046*
C2	0.51425 (13)	0.26370 (16)	0.89872 (7)	0.0337 (3)
C21	0.37448 (16)	0.3658 (2)	0.85117 (8)	0.0453 (3)
H21A	0.3406	0.4585	0.8898	0.063*
H21B	0.2834	0.2840	0.8334	0.063*
H21C	0.4076	0.4221	0.7991	0.063*
N3	0.54348 (11)	0.24577 (14)	0.98488 (6)	0.0372 (2)
C4	0.68361 (13)	0.14672 (16)	0.99900 (7)	0.0344 (3)
N4	0.75007 (12)	0.09068 (15)	1.08599 (7)	0.0416 (3)
O41	0.67792 (13)	0.13436 (17)	1.14800 (6)	0.0582 (3)
O42	0.87520 (12)	−0.00099 (17)	1.09452 (7)	0.0598 (3)
C5	0.74372 (13)	0.10265 (15)	0.92353 (7)	0.0343 (3)
H5	0.8390	0.0362	0.9176	0.041*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0286 (4)	0.0398 (5)	0.0251 (4)	0.0019 (3)	0.0023 (3)	−0.0022 (3)
C11	0.0336 (5)	0.0343 (5)	0.0262 (5)	0.0004 (4)	0.0059 (4)	−0.0009 (4)
C12	0.0362 (6)	0.0530 (7)	0.0300 (5)	−0.0047 (5)	0.0052 (4)	−0.0034 (4)
N13	0.0498 (6)	0.0659 (7)	0.0289 (5)	−0.0043 (5)	0.0042 (4)	−0.0064 (4)
C14	0.0597 (8)	0.0559 (8)	0.0290 (5)	0.0035 (6)	0.0131 (5)	−0.0010 (5)
C15	0.0469 (7)	0.0521 (7)	0.0413 (6)	0.0014 (5)	0.0213 (5)	0.0034 (5)
C16	0.0354 (6)	0.0428 (6)	0.0390 (6)	−0.0026 (4)	0.0091 (4)	−0.0004 (5)
C2	0.0300 (5)	0.0433 (6)	0.0277 (5)	0.0026 (4)	0.0036 (4)	−0.0024 (4)
C21	0.0394 (6)	0.0584 (8)	0.0369 (6)	0.0146 (5)	0.0013 (5)	0.0004 (5)
N3	0.0331 (5)	0.0508 (6)	0.0274 (4)	0.0040 (4)	0.0037 (3)	−0.0019 (4)
C4	0.0302 (5)	0.0449 (6)	0.0270 (5)	−0.0005 (4)	0.0001 (4)	0.0013 (4)
N4	0.0352 (5)	0.0567 (6)	0.0308 (5)	−0.0026 (4)	−0.0021 (4)	0.0058 (4)
O41	0.0555 (6)	0.0917 (8)	0.0270 (4)	0.0024 (5)	0.0043 (4)	0.0054 (4)
O42	0.0460 (5)	0.0805 (8)	0.0496 (6)	0.0150 (5)	−0.0043 (4)	0.0147 (5)
C5	0.0299 (5)	0.0407 (6)	0.0314 (5)	0.0021 (4)	0.0008 (4)	0.0000 (4)

Geometric parameters (Å, °)

N1—C5	1.3505 (14)	C16—H16	0.9300
N1—C2	1.3738 (13)	C2—N3	1.2960 (14)
N1—C11	1.4149 (13)	C2—C21	1.4608 (16)
C11—C12	1.3642 (16)	C21—H21A	0.9600
C11—C16	1.3696 (15)	C21—H21B	0.9600
C12—N13	1.3195 (15)	C21—H21C	0.9600
C12—H12	0.9300	N3—C4	1.3418 (14)
N13—C14	1.3181 (18)	C4—C5	1.3419 (15)
C14—C15	1.359 (2)	C4—N4	1.4089 (14)
C14—H14	0.9300	N4—O42	1.2102 (14)
C15—C16	1.3707 (17)	N4—O41	1.2165 (14)
C15—H15	0.9300	C5—H5	0.9300
C5—N1—C2	107.26 (9)	N3—C2—N1	111.18 (10)
C5—N1—C11	124.28 (9)	N3—C2—C21	124.35 (10)
C2—N1—C11	128.40 (9)	N1—C2—C21	124.46 (10)
C12—C11—C16	118.99 (10)	C2—C21—H21A	109.5
C12—C11—N1	119.95 (10)	C2—C21—H21B	109.5
C16—C11—N1	121.00 (10)	H21A—C21—H21B	109.5
N13—C12—C11	122.99 (11)	C2—C21—H21C	109.5
N13—C12—H12	118.5	H21A—C21—H21C	109.5
C11—C12—H12	118.5	H21B—C21—H21C	109.5
C14—N13—C12	117.46 (11)	C2—N3—C4	104.15 (9)
N13—C14—C15	123.66 (11)	N3—C4—C5	113.49 (10)
N13—C14—H14	118.2	N3—C4—N4	120.66 (10)
C15—C14—H14	118.2	C5—C4—N4	125.77 (11)
C14—C15—C16	118.62 (11)	O42—N4—O41	123.64 (11)
C14—C15—H15	120.7	O42—N4—C4	117.76 (11)
C16—C15—H15	120.7	O41—N4—C4	118.59 (11)
C11—C16—C15	118.27 (11)	C4—C5—N1	103.91 (9)
C11—C16—H16	120.9	C4—C5—H5	128.0
C15—C16—H16	120.9	N1—C5—H5	128.0
C5—N1—C11—C12	129.18 (12)	C5—N1—C2—C21	178.35 (12)
C2—N1—C11—C12	−47.68 (16)	C11—N1—C2—C21	−4.36 (19)
C5—N1—C11—C16	−48.21 (16)	N1—C2—N3—C4	0.08 (13)
C2—N1—C11—C16	134.93 (12)	C21—C2—N3—C4	−178.71 (12)
C16—C11—C12—N13	−0.83 (19)	C2—N3—C4—C5	0.31 (14)
N1—C11—C12—N13	−178.27 (11)	C2—N3—C4—N4	−176.59 (10)
C11—C12—N13—C14	0.3 (2)	N3—C4—N4—O42	178.79 (11)
C12—N13—C14—C15	0.2 (2)	C5—C4—N4—O42	2.28 (19)
N13—C14—C15—C16	−0.3 (2)	N3—C4—N4—O41	−0.16 (18)
C12—C11—C16—C15	0.74 (18)	C5—C4—N4—O41	−176.67 (12)
N1—C11—C16—C15	178.15 (11)	N3—C4—C5—N1	−0.57 (13)
C14—C15—C16—C11	−0.23 (19)	N4—C4—C5—N1	176.15 (11)
C5—N1—C2—N3	−0.44 (13)	C2—N1—C5—C4	0.58 (12)
C11—N1—C2—N3	176.85 (10)	C11—N1—C5—C4	−176.84 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O41^{i}	0.93	2.57	3.2872 (16)	135
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Symmetry codes: (i) −*x*+1, −*y*, −*z*+2; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*+1/2, −*y*+1/2, *z*−1/2; (iv) −*x*+2, −*y*, −*z*+2.

Fig. 1

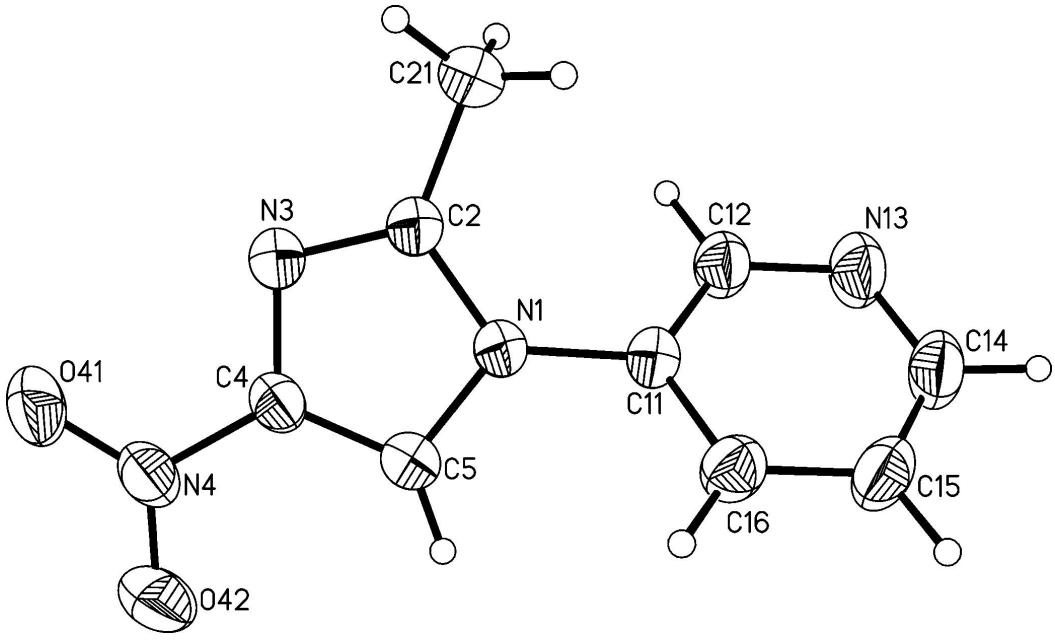

Fig. 2