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3-Methyl-1-(3-nitrophenyl)-5-phenyl-4,5-dihydro-1*H*-pyrazole

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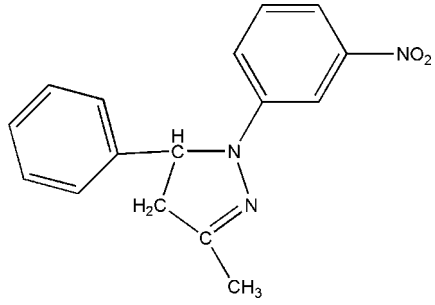
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.128; data-to-parameter ratio = 15.9.

In the title compound, $C_{16}H_{15}N_3O_2$, the planar [maximum deviation 0.156 (2) Å] pyrazoline ring is nearly coplanar with the 3-nitrophenyl group and is approximately perpendicular to the phenyl ring, making dihedral angles of 3.80 (8) and 80.58 (10)°, respectively. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure.

Related literature

For applications of pyrazoline derivatives, see: Hatheway *et al.* (1978); Mahajan *et al.* (1991); Sobczak & Pawlaczyk (1998).



Experimental

Crystal data

$C_{16}H_{15}N_3O_2$ M_r = 281.31
Monoclinic, $P2_1/n$
a = 12.0173 (4) Å
b = 7.9324 (2) Å
c = 15.4944 (5) Å
β = 99.160 (2)°
V = 1458.18 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.36 × 0.18 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
10272 measured reflections
3014 independent reflections
1648 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.128
S = 1.00
3014 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14A···O1^{i}	0.93	2.51	3.245 (2)	136

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2579).

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supporting information

Acta Cryst. (2009). E**65**, o2156 [doi:10.1107/S1600536809031390]

3-Methyl-1-(3-nitrophenyl)-5-phenyl-4,5-dihydro-1*H*-pyrazole

Jun-qiang Chen, He-ping Li, Chang-shan Huang and Jin-ying Wu

S1. Comment

The derivatives of pyrazoline are mostly used in medicine, for example as antitumor (Hatheway *et al.*, 1978), analgesic (Sobczak & Pawlaczyk, 1998), and antimicrobial (Mahajan *et al.*, 1991) agents. As part of our work, the title compound is recently synthesized in our group and its crystal structure is reported here.

The pyrazoline ring and the 3-nitrophenyl ring are nearly coplanar, making a dihedral angle of 3.80 (8)°, while the dihedral angle between the pyrazoline ring and the C1-phenyl ring is 80.58 (10)° (Fig. 1). Intermolecular weak C—H···O hydrogen bonding is present in the crystal structure (Fig. 2 and Table 1).

S2. Experimental

3-Nitrophenylhydrazine (1 mmol, 0.153 g) was dissolved in anhydrous ethanol (15 ml). The mixture was stirred for several min at 351 K, benzylideneacetone (1 mmol, 0.146 g) in ethanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized from methanol, red single crystals were obtained after 2 d.

S3. Refinement

All H atoms were positioned geometrically and refined as riding with C—H = 0.93 (aromatic), 0.97 (methylene), 0.98 (methine) and 0.96 Å (methyl), with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for the others.

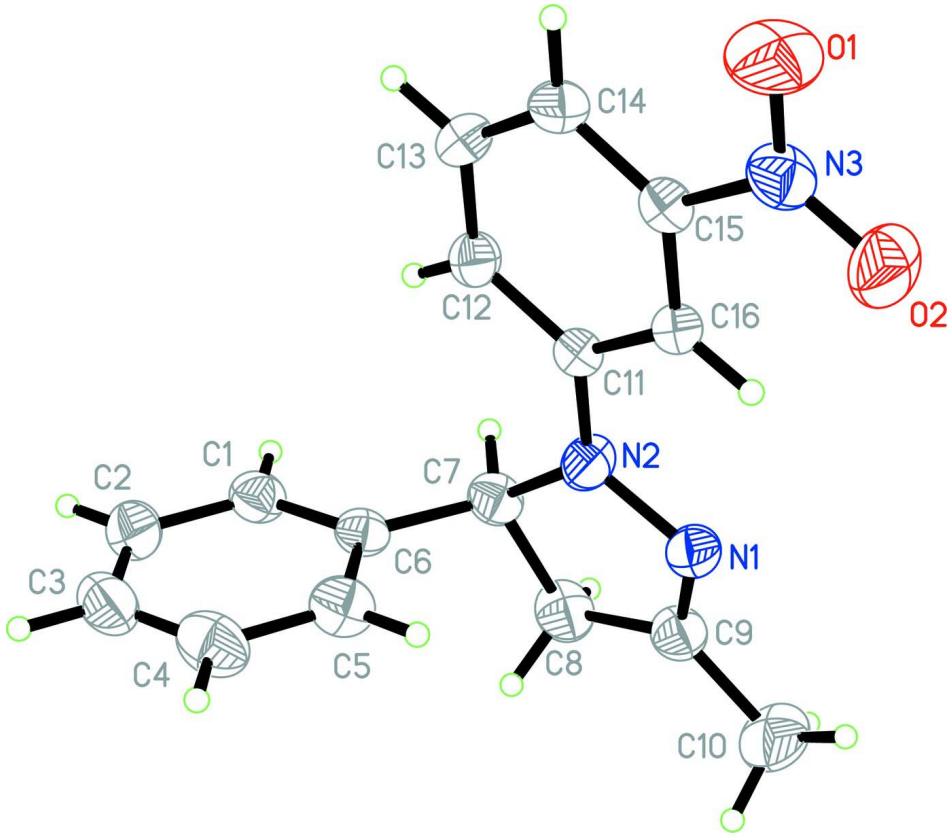

Figure 1

The molecular structure of the compound. The displacement ellipsoids are drawn at the 30% probability level.

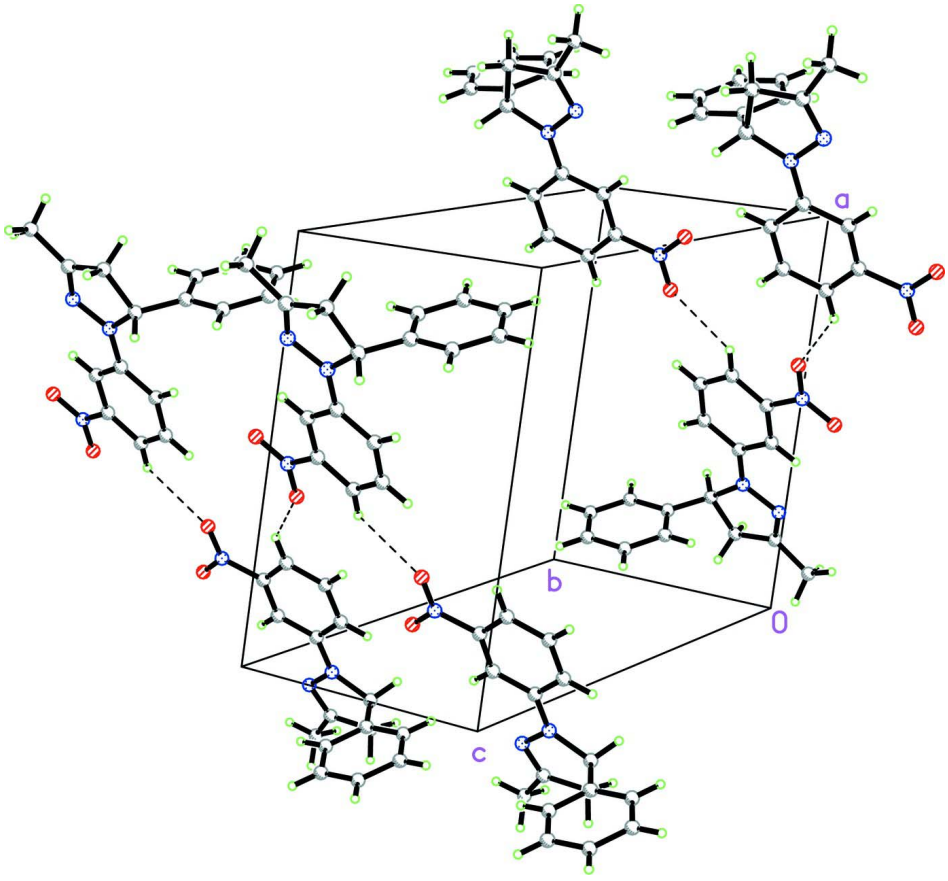

Figure 2

Packing of (I), showing the intermolecular hydrogen bonds as dashed lines.

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$M_r = 281.31$
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$c = 15.4944$ (5) Å
$\beta = 99.160$ (2)°
$V = 1458.18$ (8) Å^3
$Z = 4$
$F(000) = 592$
$D_x = 1.281$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1824 reflections
$\theta = 2.6$–$26.5°$
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Plate, red
0.36 × 0.18 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
10272 measured reflections
3014 independent reflections
1648 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.0°$
$h = -14 \rightarrow 15$
$k = -9 \rightarrow 8$
$l = -18 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.128$
$S = 1.00$
3014 reflections
190 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C11	0.40009 (13)	0.17187 (19)	0.06087 (10)	0.0433 (4)
N2	0.29773 (12)	0.18897 (16)	0.00749 (10)	0.0578 (4)
C15	0.54447 (14)	0.00161 (19)	0.13960 (10)	0.0454 (4)
C7	0.24060 (14)	0.3481 (2)	−0.02008 (11)	0.0535 (5)
H7A	0.2879	0.4156	−0.0529	0.064*
C16	0.44095 (13)	0.01314 (19)	0.08743 (10)	0.0425 (4)
H16A	0.3991	−0.0832	0.0703	0.051*
N1	0.23515 (12)	0.04742 (18)	−0.02230 (9)	0.0548 (4)
C12	0.46581 (14)	0.3125 (2)	0.08824 (11)	0.0519 (5)
H4A	0.4395	0.4197	0.0714	0.062*
C6	0.21138 (13)	0.4493 (2)	0.05545 (11)	0.0472 (4)
C13	0.56934 (14)	0.2941 (2)	0.13993 (12)	0.0561 (5)
H13A	0.6121	0.3895	0.1572	0.067*
N3	0.58488 (14)	−0.1672 (2)	0.16800 (11)	0.0619 (4)
C1	0.21969 (14)	0.6221 (2)	0.05691 (13)	0.0584 (5)
H1B	0.2456	0.6777	0.0111	0.070*
O1	0.67543 (13)	−0.17917 (18)	0.21576 (11)	0.0990 (6)
C14	0.61085 (14)	0.1383 (2)	0.16652 (11)	0.0536 (5)
H14A	0.6809	0.1259	0.2013	0.064*
O2	0.52813 (13)	−0.28871 (17)	0.14279 (11)	0.0905 (5)
C5	0.17261 (15)	0.3710 (3)	0.12393 (13)	0.0650 (5)
H5A	0.1664	0.2542	0.1241	0.078*
C8	0.13676 (16)	0.2830 (2)	−0.08217 (13)	0.0684 (6)
H8A	0.0675	0.3240	−0.0649	0.082*
H8B	0.1391	0.3168	−0.1420	0.082*

C9	0.14648 (15)	0.0972 (2)	−0.07219 (12)	0.0591 (5)
C4	0.14288 (17)	0.4619 (3)	0.19199 (14)	0.0777 (6)
H12A	0.1166	0.4065	0.2377	0.093*
C2	0.19016 (16)	0.7141 (3)	0.12524 (16)	0.0729 (6)
H2A	0.1963	0.8310	0.1255	0.088*
C3	0.15175 (17)	0.6329 (4)	0.19279 (14)	0.0777 (6)
H3A	0.1318	0.6945	0.2391	0.093*
C10	0.06227 (18)	−0.0247 (3)	−0.11675 (16)	0.0957 (8)
H10A	0.0856	−0.1375	−0.1002	0.144*
H10B	0.0567	−0.0124	−0.1789	0.144*
H10C	−0.0098	−0.0029	−0.0999	0.144*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0398 (10)	0.0436 (10)	0.0470 (10)	0.0024 (8)	0.0079 (8)	−0.0019 (8)
N2	0.0495 (9)	0.0418 (8)	0.0756 (11)	0.0045 (7)	−0.0098 (8)	−0.0003 (7)
C15	0.0460 (10)	0.0452 (10)	0.0457 (10)	0.0054 (8)	0.0097 (8)	0.0021 (8)
C7	0.0505 (11)	0.0526 (11)	0.0567 (11)	0.0095 (9)	0.0057 (9)	0.0079 (9)
C16	0.0402 (10)	0.0422 (10)	0.0451 (10)	−0.0005 (7)	0.0066 (8)	−0.0016 (7)
N1	0.0486 (9)	0.0553 (9)	0.0586 (10)	−0.0003 (7)	0.0028 (8)	−0.0044 (7)
C12	0.0524 (11)	0.0414 (10)	0.0613 (12)	0.0019 (8)	0.0072 (9)	−0.0004 (8)
C6	0.0393 (10)	0.0503 (11)	0.0509 (11)	0.0056 (8)	0.0044 (8)	0.0076 (8)
C13	0.0483 (11)	0.0520 (11)	0.0669 (13)	−0.0111 (9)	0.0059 (10)	−0.0090 (9)
N3	0.0575 (10)	0.0596 (11)	0.0673 (11)	0.0122 (9)	0.0054 (9)	0.0115 (9)
C1	0.0515 (11)	0.0548 (12)	0.0698 (13)	0.0035 (9)	0.0127 (10)	0.0041 (10)
O1	0.0680 (10)	0.0945 (12)	0.1213 (13)	0.0163 (8)	−0.0254 (10)	0.0320 (9)
C14	0.0414 (10)	0.0600 (12)	0.0576 (12)	0.0024 (9)	0.0020 (9)	−0.0024 (9)
O2	0.0924 (11)	0.0471 (8)	0.1237 (14)	0.0038 (8)	−0.0081 (10)	0.0076 (8)
C5	0.0650 (13)	0.0650 (12)	0.0661 (13)	0.0024 (10)	0.0139 (11)	0.0113 (11)
C8	0.0642 (13)	0.0779 (14)	0.0579 (13)	0.0157 (11)	−0.0064 (10)	−0.0013 (10)
C9	0.0494 (11)	0.0694 (13)	0.0555 (12)	0.0051 (10)	−0.0012 (10)	−0.0062 (10)
C4	0.0732 (15)	0.1004 (19)	0.0631 (15)	0.0073 (13)	0.0215 (12)	0.0091 (13)
C2	0.0633 (13)	0.0636 (13)	0.0913 (17)	0.0054 (11)	0.0102 (13)	−0.0148 (12)
C3	0.0609 (13)	0.1077 (19)	0.0644 (15)	0.0120 (13)	0.0094 (11)	−0.0195 (14)
C10	0.0690 (15)	0.1000 (18)	0.1050 (19)	−0.0036 (12)	−0.0269 (13)	−0.0190 (14)

Geometric parameters (Å, º)

C11—N2	1.375 (2)	N3—O2	1.2093 (18)
C11—C16	1.390 (2)	N3—O1	1.2185 (19)
C11—C12	1.393 (2)	C1—C2	1.378 (3)
N2—N1	1.3888 (18)	C1—H1B	0.9300
N2—C7	1.4678 (19)	C14—H14A	0.9300
C15—C14	1.371 (2)	C5—C4	1.371 (3)
C15—C16	1.374 (2)	C5—H5A	0.9300
C15—N3	1.468 (2)	C8—C9	1.485 (3)
C7—C6	1.506 (2)	C8—H8A	0.9700

C7—C8	1.539 (2)	C8—H8B	0.9700
C7—H7A	0.9800	C9—C10	1.488 (3)
C16—H16A	0.9300	C4—C3	1.360 (3)
N1—C9	1.275 (2)	C4—H12A	0.9300
C12—C13	1.376 (2)	C2—C3	1.370 (3)
C12—H4A	0.9300	C2—H2A	0.9300
C6—C5	1.373 (2)	C3—H3A	0.9300
C6—C1	1.374 (2)	C10—H10A	0.9600
C13—C14	1.371 (2)	C10—H10B	0.9600
C13—H13A	0.9300	C10—H10C	0.9600

N2—C11—C16	120.49 (14)	C6—C1—H1B	119.5
N2—C11—C12	120.89 (14)	C2—C1—H1B	119.5
C16—C11—C12	118.61 (15)	C15—C14—C13	117.08 (16)
C11—N2—N1	120.35 (13)	C15—C14—H14A	121.5
C11—N2—C7	126.29 (14)	C13—C14—H14A	121.5
N1—N2—C7	113.28 (13)	C4—C5—C6	121.3 (2)
C14—C15—C16	123.67 (15)	C4—C5—H5A	119.4
C14—C15—N3	118.79 (15)	C6—C5—H5A	119.4
C16—C15—N3	117.54 (15)	C9—C8—C7	103.05 (14)
N2—C7—C6	112.85 (14)	C9—C8—H8A	111.2
N2—C7—C8	100.85 (13)	C7—C8—H8A	111.2
C6—C7—C8	113.49 (13)	C9—C8—H8B	111.2
N2—C7—H7A	109.8	C7—C8—H8B	111.2
C6—C7—H7A	109.8	H8A—C8—H8B	109.1
C8—C7—H7A	109.8	N1—C9—C8	114.49 (16)
C15—C16—C11	118.59 (15)	N1—C9—C10	121.42 (18)
C15—C16—H16A	120.7	C8—C9—C10	124.09 (17)
C11—C16—H16A	120.7	C3—C4—C5	120.1 (2)
C9—N1—N2	107.90 (15)	C3—C4—H12A	120.0
C13—C12—C11	120.57 (15)	C5—C4—H12A	120.0
C13—C12—H4A	119.7	C3—C2—C1	119.8 (2)
C11—C12—H4A	119.7	C3—C2—H2A	120.1
C5—C6—C1	118.09 (17)	C1—C2—H2A	120.1
C5—C6—C7	120.62 (16)	C4—C3—C2	119.8 (2)
C1—C6—C7	121.27 (16)	C4—C3—H3A	120.1
C14—C13—C12	121.49 (16)	C2—C3—H3A	120.1
C14—C13—H13A	119.3	C9—C10—H10A	109.5
C12—C13—H13A	119.3	C9—C10—H10B	109.5
O2—N3—O1	122.49 (16)	H10A—C10—H10B	109.5
O2—N3—C15	119.20 (15)	C9—C10—H10C	109.5
O1—N3—C15	118.31 (16)	H10A—C10—H10C	109.5
C6—C1—C2	120.93 (19)	H10B—C10—H10C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C14—H14*A*···O1^{i}	0.93	2.51	3.245 (2)	136

Symmetry code: (i) −*x*+3/2, *y*+1/2, −*z*+1/2.