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3-Acetyl-1-(3-methylphenyl)-5-phenyl-1*H*-pyrazole-4-carbonitrile

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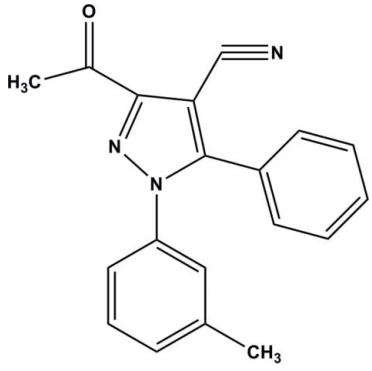
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.059; wR factor = 0.227; data-to-parameter ratio = 13.3.

In the title compound, $C_{19}H_{15}N_3O$, the central pyrazole ring makes dihedral angles of 35.52 (12) and 62.21 (11)° with the attached phenyl and methyl-substituted phenyl rings, respectively. The corresponding angle between the phenyl and methyl-substituted phenyl rings is 62.90 (11)°. In the crystal, molecules are connected by weak C—H···O hydrogen bonds, forming supramolecular chains propagating along the *a*-axis direction.

Related literature

For details and applications of pyrazole compounds, see: Kovbasyuk *et al.* (2004); Sachse *et al.* (2008); De Geest *et al.* (2007); Roy *et al.* (2008). For related structures, see: Fun *et al.* (2011*a*,*b*,*c*). For further synthetic details, see: Nassar *et al.* (2011).



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Experimental

Crystal data

$C_{19}H_{15}N_3O$
M_r = 301.34
Monoclinic, $P2_1/c$
a = 11.8544 (8) Å
b = 7.6731 (6) Å
c = 17.4048 (15) Å
β = 96.202 (6)°
V = 1573.9 (2) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 296 K
0.55 × 0.21 × 0.16 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.717, T_{max} = 0.902
8352 measured reflections
2776 independent reflections
1804 reflections with $I > 2\sigma(I)$
R_{int} = 0.091

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.227
S = 1.05
2776 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···O1^{i}	0.93	2.56	3.444 (3)	160

Symmetry code: (i) $x-1, y, z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6607).

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supplementary materials

Acta Cryst. (2012). E**68**, o485-o486 [**doi:10.1107/S160053681200181X**]

3-Acetyl-1-(3-methylphenyl)-5-phenyl-1*H*-pyrazole-4-carbonitrile

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Comment

Pyrazole-based ligands have attracted considerable attention due to their bridging nature and possibility for easy functionalization with various additional donor groups (Kovbasyuk *et al.*, 2004; Sachse *et al.*, 2008). In particular, azomethine-functionalized pyrazoles have been used extensively as ligands in the field of coordination chemistry and catalysis (De Geest *et al.*, 2007; Roy *et al.*, 2008). The crystal structures of 4-(1,3-Diphenyl-4,5-dihydro-1*H*-pyrazol-5-yl)-1,3-diphenyl- 1*H*-pyrazole, 3-Methyl-5-oxo-4-(2-phenylhydrazinylidene)-4,5- dihydro-1*H*-pyrazole-1-carbothioamide and (2*E*)-3-(1,3- Diphenyl-1*H*-pyrazol-4-yl)-1-phenylprop-2-en-1-one (Fun *et al.*, 2011*a,b,c*) have been reported from our laboratory. In continuation of our studies of pyrazole compounds, the crystal structure determination of the title compound has been undertaken.

The asymmetric unit of the title compound is shown in Fig. 1. The central pyrazole (N1,N2/C9–C11) ring makes dihedral angles of 35.52 (12) and 62.21 (11)° with the attached phenyl (C12–C17) and methyl substituted pheny (C1–C6) rings. The corresponding angle between the phenyl (C12–C17) and methyl substituted (C1–C6) phenyl rings is 62.90 (11)°.

In the crystal structure (Fig. 2), molecules are connected by weak intermolecular C—H···O (Table 1) hydrogen bonds, forming supramolecular chains propagating along the *a*-axis direction.

Experimental

The title compound was prepared according to the reported method (Nassar *et al.*, 2011). Colourless blocks were obtained by slowly evaporating from ethanol at room temperature.

Refinement

All hydrogen atoms were positioned geometrically [C–H = 0.93 or 0.96 Å] and were refined using a riding model, with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). A rotating group model was applied to the methyl groups.

Figures

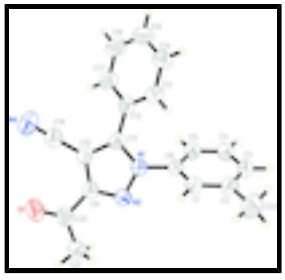

Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids.

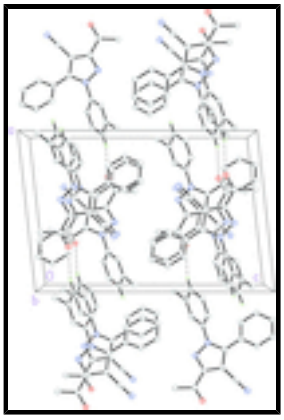

Fig. 2. The crystal packing of title compound (I).

3-Acetyl-1-(3-methylphenyl)-5-phenyl-1*H*-pyrazole-4-carbonitrile

Crystal data

$C_{19}H_{15}N_3O$	$F(000) = 632$
$M_r = 301.34$	$D_x = 1.272$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Hall symbol: -P 2ybc	Cell parameters from 1651 reflections
$a = 11.8544$ (8) Å	$\theta = 6.3$–$66.5°$
$b = 7.6731$ (6) Å	$\mu = 0.65$ mm^{-1}
$c = 17.4048$ (15) Å	$T = 296$ K
$\beta = 96.202$ (6)°	Block, colourless
$V = 1573.9$ (2) Å^3	0.55 × 0.21 × 0.16 mm
$Z = 4$	

Data collection

Bruker SMART APEXII CCD diffractometer	2776 independent reflections
Radiation source: fine-focus sealed tube	1804 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.091$
φ and ω scans	$\theta_{max} = 67.4°$, $\theta_{min} = 3.8°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	$h = -11 \rightarrow 14$
$T_{min} = 0.717$, $T_{max} = 0.902$	$k = -9 \rightarrow 8$
8352 measured reflections	$l = -19 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.059$	H-atom parameters constrained
$wR(F^2) = 0.227$	$w = 1/[\sigma^2(F_o^2) + (0.1264P)^2 + 0.0573P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2776 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
209 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}
0 restraints	Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0042 (13)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.18649 (19)	−0.0026 (3)	0.85181 (12)	0.0503 (7)
H1A	0.2331	−0.0955	0.8688	0.060*
C2	0.0758 (2)	0.0011 (3)	0.86879 (13)	0.0550 (7)
C3	0.0088 (2)	0.1432 (3)	0.84253 (14)	0.0586 (7)
H3A	−0.0662	0.1492	0.8534	0.070*
C4	0.0527 (2)	0.2746 (3)	0.80077 (14)	0.0590 (7)
H4A	0.0064	0.3677	0.7837	0.071*
C5	0.1629 (2)	0.2711 (3)	0.78384 (13)	0.0538 (6)
H5A	0.1924	0.3604	0.7559	0.065*
C6	0.22883 (19)	0.1295 (3)	0.80997 (12)	0.0461 (6)
N1	0.34545 (16)	0.1197 (2)	0.79402 (11)	0.0489 (6)
N2	0.42757 (17)	0.1217 (2)	0.85538 (11)	0.0523 (6)
C9	0.5249 (2)	0.1171 (3)	0.82443 (14)	0.0482 (6)
C10	0.5062 (2)	0.1118 (3)	0.74350 (13)	0.0496 (7)
C11	0.38934 (19)	0.1128 (2)	0.72492 (13)	0.0458 (6)
C12	0.32137 (19)	0.1023 (3)	0.64932 (13)	0.0480 (6)
C13	0.2211 (2)	0.0089 (3)	0.63772 (13)	0.0521 (6)
H13A	0.1937	−0.0474	0.6792	0.062*
C14	0.1610 (2)	−0.0016 (3)	0.56516 (14)	0.0615 (7)
H14A	0.0931	−0.0634	0.5583	0.074*
C15	0.2016 (2)	0.0791 (4)	0.50341 (16)	0.0684 (8)
H15A	0.1609	0.0724	0.4547	0.082*
C16	0.3012 (2)	0.1689 (3)	0.51313 (15)	0.0687 (8)
H16A	0.3289	0.2213	0.4708	0.082*

C17	0.3613 (2)	0.1829 (3)	0.58552 (13)	0.0588 (7)
H17A	0.4286	0.2462	0.5918	0.071*
C18	0.5895 (2)	0.1047 (3)	0.69062 (17)	0.0593 (7)
N4	0.6559 (2)	0.1033 (3)	0.64697 (16)	0.0835 (8)
C20	0.6343 (2)	0.1168 (3)	0.87444 (16)	0.0569 (7)
O1	0.72089 (16)	0.0876 (3)	0.84499 (12)	0.0754 (6)
C23	0.0279 (3)	−0.1453 (4)	0.91259 (19)	0.0857 (10)
H23A	0.0861	−0.2303	0.9263	0.129*
H23D	−0.0337	−0.1989	0.8808	0.129*
H23B	0.0008	−0.1002	0.9587	0.129*
C21	0.6332 (2)	0.1535 (4)	0.95825 (15)	0.0720 (8)
H21A	0.7093	0.1493	0.9834	0.108*
H21B	0.5877	0.0677	0.9806	0.108*
H21C	0.6018	0.2672	0.9648	0.108*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0445 (16)	0.0626 (13)	0.0437 (14)	0.0026 (10)	0.0046 (11)	0.0018 (9)
C2	0.0493 (16)	0.0690 (14)	0.0472 (13)	−0.0093 (11)	0.0078 (11)	−0.0001 (10)
C3	0.0421 (15)	0.0816 (16)	0.0528 (15)	0.0036 (11)	0.0086 (12)	−0.0018 (11)
C4	0.0484 (16)	0.0692 (14)	0.0592 (16)	0.0083 (12)	0.0045 (12)	0.0040 (11)
C5	0.0506 (15)	0.0614 (13)	0.0499 (14)	0.0022 (11)	0.0078 (11)	0.0052 (10)
C6	0.0397 (15)	0.0631 (12)	0.0358 (13)	0.0000 (9)	0.0048 (10)	−0.0027 (9)
N1	0.0401 (12)	0.0642 (11)	0.0426 (12)	0.0001 (8)	0.0054 (9)	−0.0009 (8)
N2	0.0423 (13)	0.0694 (12)	0.0443 (12)	−0.0013 (9)	0.0009 (10)	−0.0006 (8)
C9	0.0412 (15)	0.0551 (12)	0.0491 (15)	−0.0021 (9)	0.0083 (11)	−0.0004 (9)
C10	0.0449 (15)	0.0538 (12)	0.0515 (15)	0.0006 (9)	0.0112 (11)	−0.0009 (9)
C11	0.0479 (16)	0.0495 (11)	0.0413 (14)	−0.0013 (9)	0.0105 (11)	0.0015 (8)
C12	0.0470 (15)	0.0528 (12)	0.0452 (14)	0.0058 (9)	0.0100 (11)	−0.0015 (8)
C13	0.0524 (15)	0.0598 (13)	0.0446 (13)	0.0006 (11)	0.0080 (11)	−0.0033 (9)
C14	0.0573 (17)	0.0707 (15)	0.0553 (15)	0.0001 (12)	0.0007 (13)	−0.0077 (12)
C15	0.074 (2)	0.0811 (17)	0.0479 (16)	0.0099 (14)	−0.0039 (14)	−0.0015 (12)
C16	0.083 (2)	0.0761 (16)	0.0487 (16)	0.0035 (14)	0.0133 (14)	0.0132 (12)
C17	0.0654 (17)	0.0610 (14)	0.0510 (15)	−0.0043 (11)	0.0112 (12)	0.0052 (10)
C18	0.0510 (18)	0.0670 (15)	0.0610 (18)	−0.0022 (11)	0.0110 (14)	−0.0011 (11)
N4	0.0713 (18)	0.1056 (18)	0.0788 (19)	0.0031 (13)	0.0314 (15)	0.0018 (13)
C20	0.0425 (16)	0.0623 (14)	0.0655 (17)	−0.0066 (11)	0.0044 (13)	0.0035 (11)
O1	0.0429 (13)	0.0973 (14)	0.0857 (15)	−0.0028 (9)	0.0053 (11)	−0.0070 (10)
C23	0.077 (2)	0.096 (2)	0.088 (2)	−0.0160 (16)	0.0272 (18)	0.0198 (16)
C21	0.0598 (17)	0.0930 (19)	0.0603 (17)	−0.0084 (14)	−0.0071 (14)	−0.0044 (14)

Geometric parameters (Å, °)

C1—C6	1.375 (3)	C12—C13	1.384 (3)
C1—C2	1.376 (3)	C12—C17	1.398 (3)
C1—H1A	0.9300	C13—C14	1.384 (3)
C2—C3	1.396 (3)	C13—H13A	0.9300
C2—C23	1.503 (4)	C14—C15	1.372 (4)

C3—C4	1.377 (3)	C14—H14A	0.9300
C3—H3A	0.9300	C15—C16	1.362 (4)
C4—C5	1.370 (3)	C15—H15A	0.9300
C4—H4A	0.9300	C16—C17	1.383 (4)
C5—C6	1.386 (3)	C16—H16A	0.9300
C5—H5A	0.9300	C17—H17A	0.9300
C6—N1	1.441 (3)	C18—N4	1.151 (3)
N1—C11	1.362 (3)	C20—O1	1.217 (3)
N1—N2	1.365 (3)	C20—C21	1.487 (4)
N2—C9	1.325 (3)	C23—H23A	0.9600
C9—C10	1.403 (3)	C23—H23D	0.9600
C9—C20	1.482 (3)	C23—H23B	0.9600
C10—C11	1.388 (3)	C21—H21A	0.9600
C10—C18	1.422 (4)	C21—H21B	0.9600
C11—C12	1.469 (3)	C21—H21C	0.9600
C6—C1—C2	120.6 (2)	C17—C12—C11	119.2 (2)
C6—C1—H1A	119.7	C14—C13—C12	120.8 (2)
C2—C1—H1A	119.7	C14—C13—H13A	119.6
C1—C2—C3	117.9 (2)	C12—C13—H13A	119.6
C1—C2—C23	121.0 (2)	C15—C14—C13	120.0 (2)
C3—C2—C23	121.2 (2)	C15—C14—H14A	120.0
C4—C3—C2	120.7 (2)	C13—C14—H14A	120.0
C4—C3—H3A	119.6	C16—C15—C14	120.3 (2)
C2—C3—H3A	119.6	C16—C15—H15A	119.9
C5—C4—C3	121.5 (2)	C14—C15—H15A	119.9
C5—C4—H4A	119.3	C15—C16—C17	120.5 (2)
C3—C4—H4A	119.3	C15—C16—H16A	119.8
C4—C5—C6	117.5 (2)	C17—C16—H16A	119.8
C4—C5—H5A	121.3	C16—C17—C12	120.2 (2)
C6—C5—H5A	121.3	C16—C17—H17A	119.9
C1—C6—C5	121.8 (2)	C12—C17—H17A	119.9
C1—C6—N1	118.43 (19)	N4—C18—C10	178.1 (3)
C5—C6—N1	119.8 (2)	O1—C20—C9	118.5 (2)
C11—N1—N2	112.52 (19)	O1—C20—C21	123.0 (2)
C11—N1—C6	129.64 (18)	C9—C20—C21	118.4 (3)
N2—N1—C6	117.81 (19)	C2—C23—H23A	109.5
C9—N2—N1	105.08 (19)	C2—C23—H23D	109.5
N2—C9—C10	111.1 (2)	H23A—C23—H23D	109.5
N2—C9—C20	120.4 (2)	C2—C23—H23B	109.5
C10—C9—C20	128.5 (3)	H23A—C23—H23B	109.5
C11—C10—C9	106.2 (2)	H23D—C23—H23B	109.5
C11—C10—C18	126.5 (2)	C20—C21—H21A	109.5
C9—C10—C18	127.3 (2)	C20—C21—H21B	109.5
N1—C11—C10	105.15 (19)	H21A—C21—H21B	109.5
N1—C11—C12	124.6 (2)	C20—C21—H21C	109.5
C10—C11—C12	130.2 (2)	H21A—C21—H21C	109.5
C13—C12—C17	118.2 (2)	H21B—C21—H21C	109.5
C13—C12—C11	122.6 (2)		

C6—C1—C2—C3	−0.3 (3)	C6—N1—C11—C10	177.65 (19)
C6—C1—C2—C23	178.3 (2)	N2—N1—C11—C12	177.50 (17)
C1—C2—C3—C4	0.2 (3)	C6—N1—C11—C12	−4.4 (3)
C23—C2—C3—C4	−178.4 (3)	C9—C10—C11—N1	0.4 (2)
C2—C3—C4—C5	−0.3 (4)	C18—C10—C11—N1	179.7 (2)
C3—C4—C5—C6	0.4 (4)	C9—C10—C11—C12	−177.45 (19)
C2—C1—C6—C5	0.4 (3)	C18—C10—C11—C12	1.9 (3)
C2—C1—C6—N1	179.76 (18)	N1—C11—C12—C13	−36.0 (3)
C4—C5—C6—C1	−0.5 (3)	C10—C11—C12—C13	141.5 (2)
C4—C5—C6—N1	−179.78 (19)	N1—C11—C12—C17	146.9 (2)
C1—C6—N1—C11	119.3 (2)	C10—C11—C12—C17	−35.6 (3)
C5—C6—N1—C11	−61.3 (3)	C17—C12—C13—C14	−1.1 (3)
C1—C6—N1—N2	−62.6 (2)	C11—C12—C13—C14	−178.3 (2)
C5—C6—N1—N2	116.7 (2)	C12—C13—C14—C15	0.9 (4)
C11—N1—N2—C9	0.4 (2)	C13—C14—C15—C16	0.3 (4)
C6—N1—N2—C9	−177.99 (17)	C14—C15—C16—C17	−1.3 (4)
N1—N2—C9—C10	−0.1 (2)	C15—C16—C17—C12	1.1 (4)
N1—N2—C9—C20	−179.71 (19)	C13—C12—C17—C16	0.2 (3)
N2—C9—C10—C11	−0.2 (2)	C11—C12—C17—C16	177.4 (2)
C20—C9—C10—C11	179.4 (2)	N2—C9—C20—O1	169.6 (2)
N2—C9—C10—C18	−179.5 (2)	C10—C9—C20—O1	−9.9 (3)
C20—C9—C10—C18	0.0 (4)	N2—C9—C20—C21	−10.8 (3)
N2—N1—C11—C10	−0.5 (2)	C10—C9—C20—C21	169.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3A···O1^{i}	0.93	2.56	3.444 (3)	160

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1

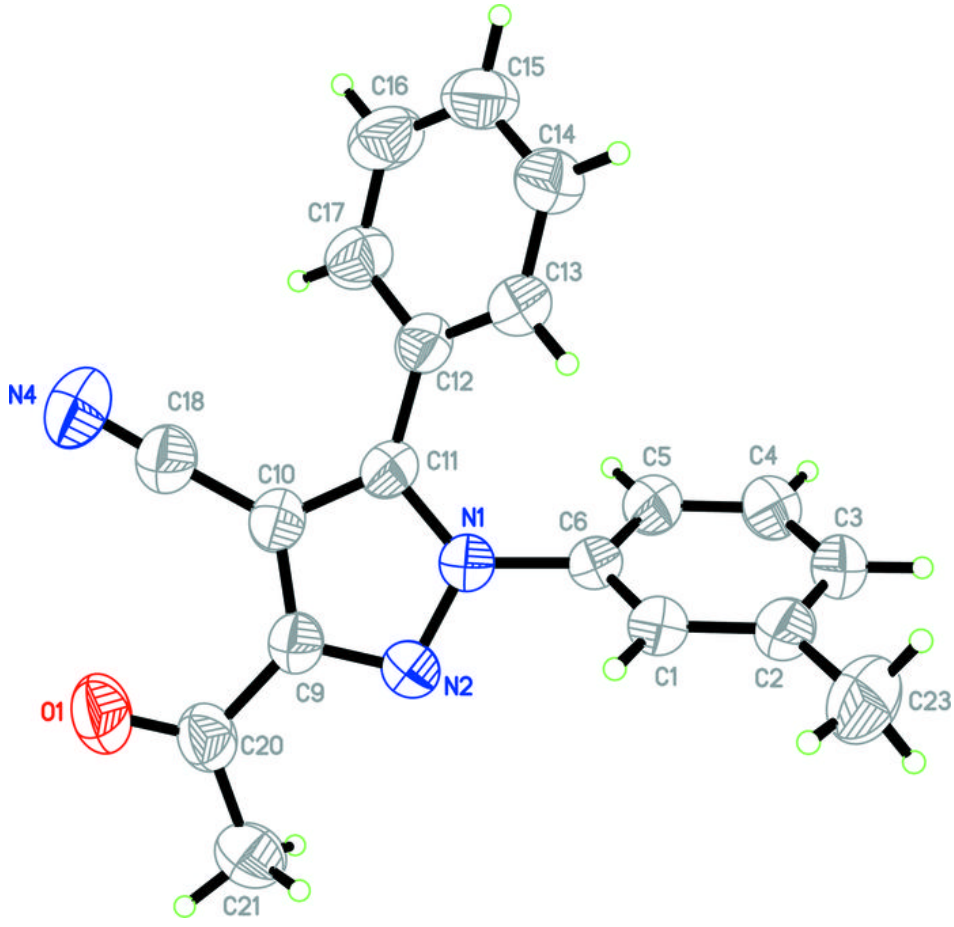
C15
C16
C14
C17
C13
C12
N4
C18
C10
C11
C5
C4
N1
C6
C3
C9
N2
C1
C2
C23
O1
C20
C21

Fig. 2