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(4*E*)-1,5-Dimethyl-4-[(5-nitrofuran-2-yl)methyleneamino]-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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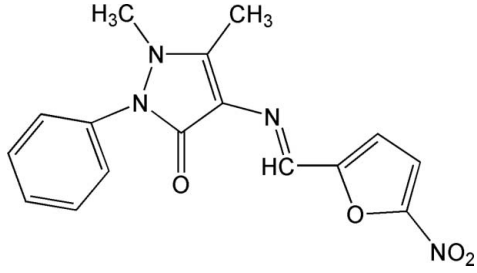
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.055; wR factor = 0.154; data-to-parameter ratio = 12.4.

In the title compound, $C_{16}H_{14}N_4O_4$, the central C_3N_2 ring is planar, with an r.m.s. deviation of 0.0253 (2) Å for the five fitted atoms, and forms dihedral angles of 5.66 (6) and 46.54 (5)°, respectively, with the (5-nitrofuran-2-yl)methylene and phenyl groups. The molecules adopt a layered arrangement, with the nitro groups accepting C—H···O interactions.

Related literature

For related literature, see: Kahwa *et al.* (1986); Santos *et al.* (2001).



Experimental

Crystal data

$C_{16}H_{14}N_4O_4$
M_r = 326.31
Monoclinic, $P2_1/n$
a = 7.0722 (14) Å
b = 7.8143 (16) Å
c = 27.917 (6) Å
β = 91.75 (3)°
V = 1542.1 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.10 × 0.08 × 0.04 mm

Data collection

Rigaku Saturn CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.990, T_{max} = 0.996
9081 measured reflections
2723 independent reflections
2175 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.154
S = 1.17
2723 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.93	2.49	3.407 (3)	170

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2210).

References

Kahwa, I. A., Selbin, J., Hsieh, T. C.-Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **118**, 179–185.
Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 838–844.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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(4*E*)-1,5-Dimethyl-4-[(5-nitrofuran-2-yl)methyleneamino]-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Z.-L. Jing, M. Yu and C. Shen

Comment

Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*,2001). As part of an investigation of the coordination properties of Shiff bases functioning as ligands, we report the synthesis and structure of the title compound.

The molecular structure (Figure 1) has expected geometric parameters. The central chromophore (C6–C8/N3/N4) is planar, with an r.m.s. deviation for the fitted atoms of 0.0253 (2) Å. The 5-nitrofuran-2-carbaldehyde group (C1–C4/O3) and phenyl ring (C11–C16) are also planar, with r.m.s. deviations of 0.0013 (3) and 0.0084 (4) Å, respectively. The dihedral angles between these latter two planes and the plane through the C6–C8/N3/N4 ring are 5.66 (6)° and 46.54 (5)°, respectively, while the C1–C4/O3 and C11–C16 planes form an angle of 46.97 (4) Å. The molecules adopt a layered arrangement (Figure 2), with C—H···O interactions formed to the NO_2 group.

Experimental

An anhydrous ethanol solution (50 ml) of 5-nitrofuran-2-carbaldehyde (1.41 g,10 mmol) was added to an anhydrous ethanol solution (50 ml) of 4-amino-1,2-dihydro-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) and the mixture was stirred at 350 K for 6 h under N_2, forming a red solution. The solvent was removed and the residue was recrystallized from anhydrous ethanol then dried *in vacuo* to give the title compound in 88% yield. Red single crystals suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Refinement

H atoms were included in calculated positions, with C—H = 0.93 (aromatic) or 0.96 Å (methyl), and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(aromatic C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate about their local threefold axes.

Figures

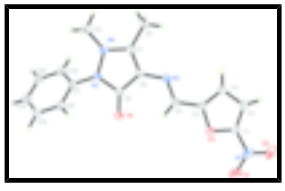

Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

Fig. 2. Packing view along the *c*-axis. Dashed lines denote C—H···O interactions.

(4E)-1,5-Dimethyl-4-[(5-nitrofuran-2-yl)methyleneamino]-2-phenyl-1*H*-pyrazol- 3(2*H*)-one

Crystal data

$C_{16}H_{14}N_4O_4$	F_{000} = 680
M_r = 326.31	D_x = 1.406 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3365 reflections
a = 7.0722 (14) Å	θ = 2.6–27.9º
b = 7.8143 (16) Å	μ = 0.10 mm^{-1}
c = 27.917 (6) Å	T = 293 (2) K
β = 91.75 (3)º	Block, red
V = 1542.1 (6) Å^3	0.10 × 0.08 × 0.04 mm
Z = 4	

Data collection

Rigaku Saturn CCD diffractometer	2723 independent reflections
Radiation source: rotating anode	2175 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.047
T = 293(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)	h = −8→8
T_{min} = 0.990, T_{max} = 0.996	k = −9→9
9081 measured reflections	l = −33→25

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.055	H-atom parameters constrained
$wR(F^2)$ = 0.154	$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 0.2344P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.17	$(\Delta/\sigma)_{max}$ = 0.005
2723 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
219 parameters	$\Delta\rho_{min}$ = −0.32 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 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 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}
O1	0.6509 (3)	0.9463 (2)	1.07595 (9)	0.0716 (7)
O2	0.5483 (3)	0.8358 (3)	1.14189 (8)	0.0708 (7)
O3	0.7068 (2)	0.64324 (18)	1.03917 (6)	0.0381 (4)
O4	0.8983 (3)	0.4723 (2)	0.88083 (6)	0.0466 (5)
N1	0.6087 (3)	0.8236 (3)	1.10124 (9)	0.0507 (6)
N2	0.7851 (3)	0.2637 (2)	0.97211 (6)	0.0353 (5)
N3	0.9515 (3)	0.1961 (2)	0.85268 (7)	0.0373 (5)
N4	0.9140 (3)	0.0287 (2)	0.86905 (7)	0.0370 (5)
C1	0.6319 (3)	0.6556 (3)	1.08303 (8)	0.0374 (6)
C2	0.5889 (4)	0.5012 (3)	1.10137 (9)	0.0423 (6)
H2	0.5368	0.4791	1.1309	0.051*
C3	0.6386 (4)	0.3810 (3)	1.06675 (8)	0.0406 (6)
H3	0.6253	0.2629	1.0688	0.049*
C4	0.7105 (3)	0.4706 (3)	1.02929 (8)	0.0342 (5)
C5	0.7813 (3)	0.4231 (3)	0.98341 (8)	0.0350 (6)
H5	0.8235	0.5060	0.9624	0.042*
C6	0.8485 (3)	0.2121 (3)	0.92785 (8)	0.0327 (5)
C7	0.8621 (3)	0.0419 (3)	0.91528 (8)	0.0345 (5)
C8	0.8995 (3)	0.3150 (3)	0.88740 (8)	0.0341 (5)
C9	0.8329 (4)	−0.1113 (3)	0.94581 (10)	0.0501 (7)
H9A	0.7707	−0.1991	0.9271	0.075*
H9B	0.7561	−0.0810	0.9723	0.075*
H9C	0.9532	−0.1528	0.9577	0.075*
C10	1.0214 (4)	−0.1151 (3)	0.84999 (10)	0.0463 (6)
H10A	1.1541	−0.0966	0.8562	0.069*
H10B	0.9973	−0.1237	0.8160	0.069*
H10C	0.9833	−0.2192	0.8652	0.069*
C11	0.9627 (3)	0.2297 (3)	0.80267 (8)	0.0366 (6)
C12	0.8539 (4)	0.1400 (3)	0.76909 (9)	0.0450 (6)
H12	0.7689	0.0572	0.7789	0.054*
C13	0.8724 (4)	0.1746 (3)	0.72060 (9)	0.0524 (7)
H13	0.8021	0.1126	0.6979	0.063*
C14	0.9938 (4)	0.2999 (3)	0.70615 (10)	0.0535 (7)

H14	1.0057	0.3226	0.6737	0.064*
C15	1.0986 (4)	0.3926 (3)	0.73983 (10)	0.0520 (7)
H15	1.1781	0.4797	0.7299	0.062*
C16	1.0858 (4)	0.3564 (3)	0.78811 (9)	0.0453 (6)
H16	1.1591	0.4166	0.8106	0.054*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0948 (18)	0.0325 (11)	0.0871 (16)	−0.0013 (10)	−0.0040 (13)	−0.0034 (10)
O2	0.0737 (15)	0.0753 (15)	0.0635 (14)	0.0122 (11)	0.0050 (12)	−0.0309 (11)
O3	0.0455 (10)	0.0303 (8)	0.0387 (9)	−0.0018 (7)	0.0023 (7)	0.0004 (7)
O4	0.0676 (13)	0.0290 (9)	0.0437 (10)	−0.0025 (8)	0.0109 (9)	0.0037 (7)
N1	0.0510 (14)	0.0420 (13)	0.0585 (15)	0.0044 (10)	−0.0068 (12)	−0.0146 (11)
N2	0.0389 (11)	0.0359 (11)	0.0311 (10)	−0.0004 (8)	0.0016 (8)	0.0018 (8)
N3	0.0514 (12)	0.0288 (10)	0.0322 (10)	−0.0041 (9)	0.0088 (9)	0.0047 (8)
N4	0.0469 (12)	0.0254 (10)	0.0391 (11)	−0.0016 (8)	0.0064 (9)	0.0022 (8)
C1	0.0384 (13)	0.0385 (13)	0.0355 (13)	−0.0003 (10)	0.0006 (10)	−0.0060 (10)
C2	0.0474 (15)	0.0443 (14)	0.0355 (13)	−0.0001 (11)	0.0053 (11)	0.0018 (10)
C3	0.0508 (15)	0.0318 (12)	0.0393 (14)	0.0000 (10)	0.0052 (11)	0.0049 (10)
C4	0.0355 (13)	0.0289 (11)	0.0381 (13)	0.0008 (9)	−0.0019 (10)	−0.0010 (9)
C5	0.0363 (14)	0.0362 (13)	0.0324 (12)	−0.0016 (10)	0.0002 (10)	0.0041 (9)
C6	0.0351 (13)	0.0296 (12)	0.0337 (12)	−0.0006 (9)	0.0032 (10)	0.0023 (9)
C7	0.0363 (13)	0.0335 (12)	0.0337 (12)	−0.0011 (10)	0.0013 (10)	0.0025 (9)
C8	0.0382 (13)	0.0292 (12)	0.0351 (12)	−0.0024 (9)	0.0020 (10)	0.0007 (9)
C9	0.0706 (19)	0.0340 (13)	0.0461 (15)	−0.0027 (12)	0.0076 (13)	0.0088 (11)
C10	0.0572 (17)	0.0329 (13)	0.0492 (15)	0.0031 (11)	0.0075 (12)	−0.0042 (11)
C11	0.0406 (14)	0.0360 (12)	0.0337 (12)	0.0025 (10)	0.0091 (10)	0.0028 (9)
C12	0.0497 (16)	0.0396 (14)	0.0457 (15)	−0.0061 (11)	0.0042 (12)	0.0021 (11)
C13	0.0683 (19)	0.0502 (15)	0.0385 (14)	0.0023 (14)	−0.0031 (13)	−0.0041 (12)
C14	0.073 (2)	0.0522 (16)	0.0368 (14)	0.0094 (14)	0.0157 (13)	0.0059 (12)
C15	0.0587 (18)	0.0511 (15)	0.0473 (16)	−0.0062 (13)	0.0207 (13)	0.0080 (12)
C16	0.0486 (16)	0.0461 (14)	0.0415 (14)	−0.0078 (12)	0.0072 (12)	0.0018 (11)

Geometric parameters (Å, °)

O1—N1	1.233 (3)	C6—C7	1.380 (3)
O2—N1	1.229 (3)	C6—C8	1.441 (3)
O3—C1	1.352 (3)	C7—C9	1.488 (3)
O3—C4	1.378 (3)	C9—H9A	0.960
O4—C8	1.242 (3)	C9—H9B	0.960
N1—C1	1.419 (3)	C9—H9C	0.960
N2—C5	1.285 (3)	C10—H10A	0.960
N2—C6	1.387 (3)	C10—H10B	0.960
N3—C8	1.400 (3)	C10—H10C	0.960
N3—N4	1.413 (2)	C11—C12	1.385 (3)
N3—C11	1.425 (3)	C11—C16	1.388 (3)
N4—C7	1.357 (3)	C12—C13	1.390 (4)
N4—C10	1.466 (3)	C12—H12	0.930

C1—C2	1.349 (3)	C13—C14	1.371 (4)
C2—C3	1.400 (4)	C13—H13	0.930
C2—H2	0.930	C14—C15	1.384 (4)
C3—C4	1.369 (3)	C14—H14	0.930
C3—H3	0.930	C15—C16	1.383 (4)
C4—C5	1.438 (3)	C15—H15	0.930
C5—H5	0.930	C16—H16	0.930
C1—O3—C4	105.17 (17)	O4—C8—C6	131.8 (2)
O2—N1—O1	124.4 (2)	N3—C8—C6	104.42 (19)
O2—N1—C1	116.8 (2)	C7—C9—H9A	109.5
O1—N1—C1	118.8 (2)	C7—C9—H9B	109.5
C5—N2—C6	120.64 (19)	H9A—C9—H9B	109.5
C8—N3—N4	109.55 (18)	C7—C9—H9C	109.5
C8—N3—C11	125.39 (19)	H9A—C9—H9C	109.5
N4—N3—C11	120.27 (18)	H9B—C9—H9C	109.5
C7—N4—N3	107.26 (17)	N4—C10—H10A	109.5
C7—N4—C10	124.19 (19)	N4—C10—H10B	109.5
N3—N4—C10	119.18 (19)	H10A—C10—H10B	109.5
C2—C1—O3	112.3 (2)	N4—C10—H10C	109.5
C2—C1—N1	131.4 (2)	H10A—C10—H10C	109.5
O3—C1—N1	116.3 (2)	H10B—C10—H10C	109.5
C1—C2—C3	105.9 (2)	C12—C11—C16	120.2 (2)
C1—C2—H2	127.1	C12—C11—N3	121.6 (2)
C3—C2—H2	127.1	C16—C11—N3	118.2 (2)
C4—C3—C2	107.0 (2)	C11—C12—C13	119.6 (2)
C4—C3—H3	126.5	C11—C12—H12	120.2
C2—C3—H3	126.5	C13—C12—H12	120.2
C3—C4—O3	109.8 (2)	C14—C13—C12	120.3 (2)
C3—C4—C5	134.1 (2)	C14—C13—H13	119.9
O3—C4—C5	116.1 (2)	C12—C13—H13	119.9
N2—C5—C4	118.6 (2)	C13—C14—C15	120.1 (3)
N2—C5—H5	120.7	C13—C14—H14	120.0
C4—C5—H5	120.7	C15—C14—H14	120.0
C7—C6—N2	122.2 (2)	C16—C15—C14	120.4 (3)
C7—C6—C8	108.5 (2)	C16—C15—H15	119.8
N2—C6—C8	129.1 (2)	C14—C15—H15	119.8
N4—C7—C6	109.74 (19)	C15—C16—C11	119.4 (2)
N4—C7—C9	122.0 (2)	C15—C16—H16	120.3
C6—C7—C9	128.2 (2)	C11—C16—H16	120.3
O4—C8—N3	123.7 (2)		
C8—N3—N4—C7	−7.3 (2)	C10—N4—C7—C9	−27.0 (3)
C11—N3—N4—C7	−164.1 (2)	N2—C6—C7—N4	175.44 (19)
C8—N3—N4—C10	−155.3 (2)	C8—C6—C7—N4	−1.4 (3)
C11—N3—N4—C10	48.0 (3)	N2—C6—C7—C9	−6.5 (4)
C4—O3—C1—C2	0.2 (3)	C8—C6—C7—C9	176.7 (2)
C4—O3—C1—N1	−178.63 (19)	N4—N3—C8—O4	−172.3 (2)
O2—N1—C1—C2	4.5 (4)	C11—N3—C8—O4	−17.0 (4)
O1—N1—C1—C2	−176.1 (3)	N4—N3—C8—C6	6.2 (2)

O2—N1—C1—O3	−177.0 (2)	C11—N3—C8—C6	161.5 (2)
O1—N1—C1—O3	2.5 (3)	C7—C6—C8—O4	175.4 (2)
O3—C1—C2—C3	−0.3 (3)	N2—C6—C8—O4	−1.2 (4)
N1—C1—C2—C3	178.2 (2)	C7—C6—C8—N3	−3.0 (2)
C1—C2—C3—C4	0.4 (3)	N2—C6—C8—N3	−179.6 (2)
C2—C3—C4—O3	−0.3 (3)	C8—N3—C11—C12	−120.2 (3)
C2—C3—C4—C5	−178.5 (2)	N4—N3—C11—C12	32.7 (3)
C1—O3—C4—C3	0.1 (2)	C8—N3—C11—C16	59.5 (3)
C1—O3—C4—C5	178.64 (19)	N4—N3—C11—C16	−147.6 (2)
C6—N2—C5—C4	178.65 (19)	C16—C11—C12—C13	1.7 (4)
C3—C4—C5—N2	−0.8 (4)	N3—C11—C12—C13	−178.6 (2)
O3—C4—C5—N2	−178.96 (19)	C11—C12—C13—C14	−1.8 (4)
C5—N2—C6—C7	177.2 (2)	C12—C13—C14—C15	0.0 (4)
C5—N2—C6—C8	−6.6 (4)	C13—C14—C15—C16	1.9 (4)
N3—N4—C7—C6	5.3 (3)	C14—C15—C16—C11	−1.9 (4)
C10—N4—C7—C6	151.2 (2)	C12—C11—C16—C15	0.1 (4)
N3—N4—C7—C9	−172.9 (2)	N3—C11—C16—C15	−179.6 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.93	2.49	3.407 (3)	170

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1

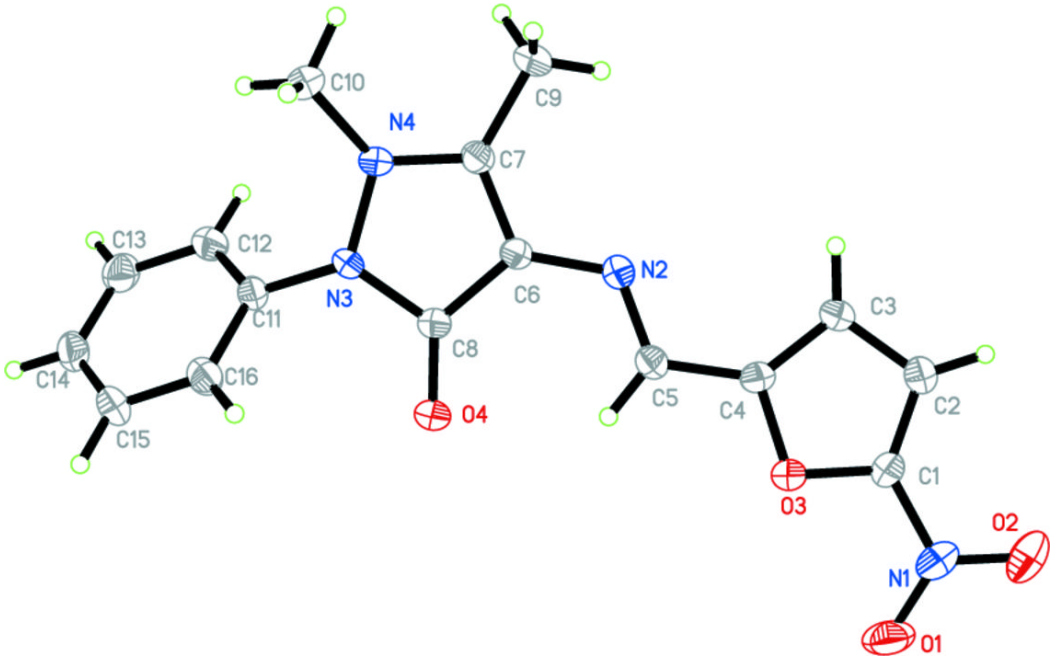
C10
C9
N4
C7
C13
C12
N3
C6
N2
C3
C11
C8
C14
C16
O4
C5
C4
C2
C15
O3
C1
O2
N1
O1

Fig. 2