

5-(2-Furyl)-3-methyl-1-(4-nitrophenyl)-2-pyrazoline

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Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.036
 wR factor = 0.091
Data-to-parameter ratio = 8.1

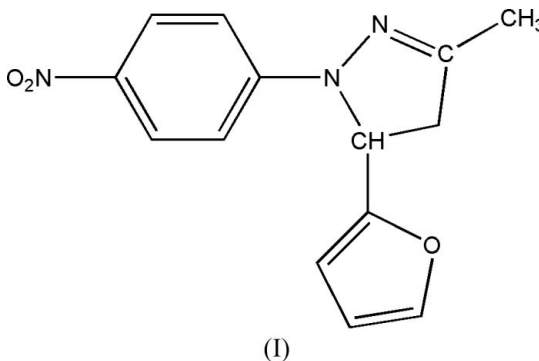
For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $\text{C}_{14}\text{H}_{13}\text{N}_3\text{O}_3$, the pyrazoline ring and the 4-nitrophenyl group are nearly coplanar, whereas the furyl and pyrazoline rings are roughly perpendicular. The occurrence of weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions results in the formation of an $R_3^3(15)$ ring.

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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 research, we have synthesized the new pyrazoline compound, (I).



The 4-nitrophenyl group and the pyrazoline ring are almost coplanar, making a dihedral angle of 3.52 (14) $^\circ$, while the furyl ring is nearly perpendicular to the pyrazoline ring, with a dihedral angle of 89.61 (15) $^\circ$. The dihedral angle between the furyl ring and the 4-nitrophenyl group is 87.93 (14) $^\circ$. The $\text{N}2-\text{N}3$ bond length [1.400 (3) Å] agrees with that of a single bond (Burke-Laing & Laing, 1976).

An interesting feature is the occurrence of weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds involving two O atoms of the nitro group, leading to the formation of an $R_3^3(15)$ graph-set motif (Etter *et al.*, 1990) (Table 1 and Fig. 2).

Experimental

4-Nitrophenylhydrazine (1 mmol, 0.153 g) was dissolved in anhydrous methanol (15 ml), and H_2SO_4 (98%, 0.5 ml) was added. The mixture was stirred for several minutes at 351 K, furylideneacetone (1 mmol, 0.136 g) in methanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized from dichloromethane, brown single crystals of (I) being obtained after 1 d.

Crystal data

$C_{14}H_{13}N_3O_3$
 $M_r = 271.27$
 Monoclinic, $P2_1$
 $a = 8.1504 (13) \text{ \AA}$
 $b = 8.4608 (14) \text{ \AA}$
 $c = 9.6959 (16) \text{ \AA}$
 $\beta = 98.036 (2)^\circ$
 $V = 662.05 (19) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.361 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.10 \text{ mm}^{-1}$
 $T = 298 (2) \text{ K}$
 Block, brown
 $0.27 \times 0.23 \times 0.20 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan *SADABS* (Bruker, 2000)
 $T_{\min} = 0.974$, $T_{\max} = 0.981$

5482 measured reflections
 1470 independent reflections
 1047 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\text{max}} = 26.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.091$
 $S = 1.00$
 1470 reflections
 182 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.0117P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.10 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C2-H2\cdots O3^i$	0.93	2.51	3.233 (4)	135
$C10-H10\cdots O2^{ii}$	0.98	2.47	3.369 (4)	153

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

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 \AA (methyl), with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{CH or CH}_2)$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{CH}_3)$. In the absence of significant anomalous scattering, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-III* (Burnett & Johnson, 1996); *ORTEP-3 for Windows* (Farrugia, 1997); *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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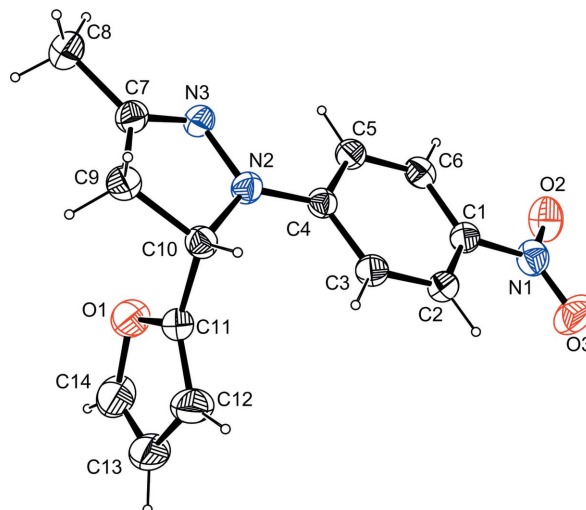


Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

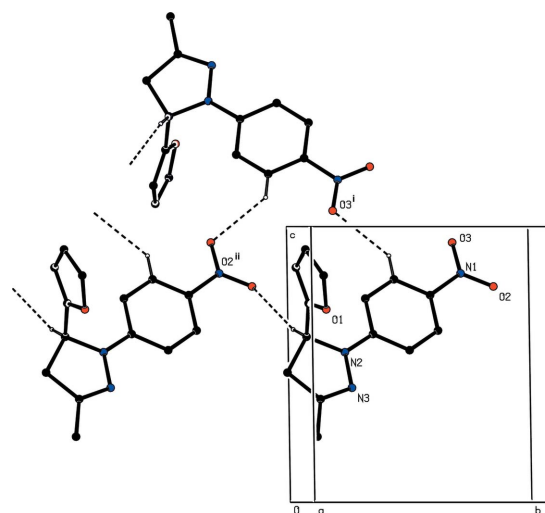


Figure 2

Partial packing view, showing the formation of a pseudo-trimer through $C-H\cdots O$ hydrogen bonding. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $-x, y - \frac{1}{2}, 2 - z$; (ii) $x, y - 1, z$].

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