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3-(4-Fluorophenyl)-1-phenyl-5-(*p*-tolyl)-2-pyrazoline

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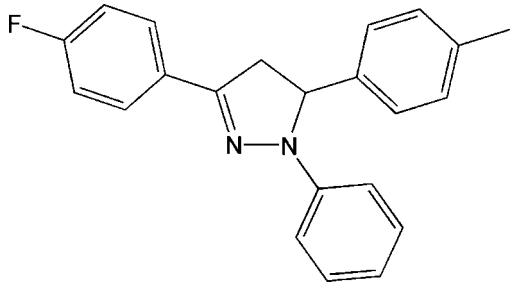
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.061; wR factor = 0.222; data-to-parameter ratio = 13.5.

The title compound, $C_{22}H_{19}FN_2$, was prepared by reacting phenylhydrazine with 1-(4-fluorophenyl)-3-(*p*-tolyl)prop-2-enone. The pyrazoline ring forms a dihedral angle of 5.2 (3)° with the phenyl ring, 8.8 (1)° with the fluorophenyl ring and 80.7 (2)° with the *p*-tolyl ring.

Related literature

For literature on the properties of pyrazoline derivatives, see: Dhal *et al.* (1975); Fahrni *et al.* (2003); Lombardino & Ottemes (1981); Rawal *et al.* (1963); Rurack *et al.* (2000); Wiley *et al.* (1958). Two crystal structures closely related to that of the title compound were previously reported (Foces-Foces *et al.*, 2001; Guo *et al.*, 2006).



Experimental

Crystal data

$C_{22}H_{19}FN_2$
M_r = 330.39
Monoclinic, $P2_1/c$
a = 20.050 (2) Å
b = 5.588 (3) Å
c = 16.480 (2) Å
β = 108.913 (19)°
V = 1747 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 294 (2) K
0.30 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.976, T_{max} = 0.984
8200 measured reflections
3081 independent reflections
1371 reflections with $I > 2\sigma(I)$
R_{int} = 0.089

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.222
S = 0.91
3081 reflections
228 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2102).

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supplementary materials

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3-(4-Fluorophenyl)-1-phenyl-5-(*p*-tolyl)-2-pyrazoline

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Comment

As important and useful five-membered heterocyclic compounds, pyrazoline and its derivatives were found to possess anti-viral (Rawal *et al.*, 1963), antifungal (Dhal *et al.*, 1975), and immunosuppressive (Lombardino & Ottemes, 1981) properties. Several 1,3,5-triaryl-2-pyrazolines were also used as scintillation solutes (Wiley *et al.*, 1958). Here, we report the crystal structure of the title compound, a functionalized pyrazoline (Fig. 1). All bond lengths and angles fall in the normal ranges (Rurack *et al.*, 2000; Fahrni *et al.*, 2003; Guo *et al.*, 2006; Foces-Foces *et al.*, 2001). The mean plane of the pyrazoline ring, N1/N2/C7/C8/C9 makes dihedral angles of 80.7 (2), 8.8 (1) and 5.2 (3)° with the benzene ring C10/C11/C12/C13/C14/C15, the benzene ring C1/C2/C3/C4/C5/C6, and the phenyl ring C17/C18/C19/C20/C21/C22, respectively.

Experimental

Amounts of 1-(4-fluorophenyl)-3-(*p*-tolyl)-2-propenone (0.02 mol) and phenylhydrazine (0.02 mol) were mixed in 99.5% acetic acid (40 ml) and refluxed for 6 h. Then, the mixture was poured into ice-water to afford yellow solids. The solids were filtrated and washed with water until the pH of solution was *ca*. 7. Yellow single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from EtOH at room temperature.

Refinement

H atoms were placed geometrically and allowed to ride on their parent atoms, with C—H distances constrained to 0.93 (aromatic CH), 0.97 (methylene CH_2) aor 0.98 Å (methine CH), and with U_{iso}(H) = 1.2U_{eq}(carrier C).

Figures

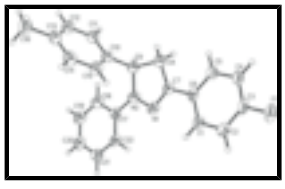


Fig. 1. The molecular structure and atom-labeling scheme for the title molecule, with displacement ellipsoids drawn at the 30% probability level.

3-(4-Fluorophenyl)-1-phenyl-5-(*p*-tolyl)-2-pyrazoline

Crystal data

$C_{22}H_{19}FN_2$	F_{000} = 696
M_r = 330.39	D_x = 1.256 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å

Hall symbol: -P 2ybc	Cell parameters from 1300 reflections
a = 20.050 (2) Å	θ = 2.5–22.9º
b = 5.588 (3) Å	μ = 0.08 mm^{-1}
c = 16.480 (2) Å	T = 294 (2) K
β = 108.913 (19)º	Block, yellow
V = 1747 (4) $Å^3$	0.30 × 0.24 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3081 independent reflections
Radiation source: fine-focus sealed tube	1371 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.089
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.1º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −23→23
T_{min} = 0.976, T_{max} = 0.984	k = −6→5
8200 measured reflections	l = −16→19

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.061	$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.222	$(\Delta/\sigma)_{max} < 0.001$
S = 0.91	$\Delta\rho_{max}$ = 0.17 e $Å^{-3}$
3081 reflections	$\Delta\rho_{min}$ = −0.19 e $Å^{-3}$
228 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.011 (3)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
F1	0.47935 (9)	0.2677 (5)	0.44618 (15)	0.0950 (9)
N1	0.67472 (13)	0.4524 (6)	0.21806 (17)	0.0555 (9)
N2	0.72962 (13)	0.4153 (6)	0.18606 (18)	0.0629 (9)
C1	0.57510 (17)	0.4651 (7)	0.3086 (2)	0.0578 (10)
H1	0.5744	0.5909	0.2714	0.069*
C2	0.52539 (17)	0.4577 (7)	0.3500 (2)	0.0616 (11)
H2	0.4909	0.5754	0.3405	0.074*
C3	0.52810 (16)	0.2733 (8)	0.4051 (2)	0.0640 (12)

C4	0.57658 (18)	0.0955 (8)	0.4198 (2)	0.0663 (11)
H4	0.5765	−0.0293	0.4571	0.080*
C5	0.62618 (17)	0.1041 (7)	0.3778 (2)	0.0576 (10)
H5	0.6601	−0.0156	0.3875	0.069*
C6	0.62597 (15)	0.2896 (7)	0.3213 (2)	0.0505 (9)
C7	0.67859 (15)	0.2964 (7)	0.2776 (2)	0.0505 (9)
C8	0.73981 (17)	0.1281 (7)	0.2917 (2)	0.0621 (11)
H8A	0.7238	−0.0321	0.2721	0.074*
H8B	0.7682	0.1217	0.3518	0.074*
C9	0.78135 (16)	0.2389 (7)	0.2373 (2)	0.0564 (10)
H9	0.7900	0.1168	0.1992	0.068*
C10	0.85006 (16)	0.3557 (7)	0.2878 (2)	0.0541 (10)
C11	0.91418 (18)	0.2616 (7)	0.2897 (2)	0.0645 (11)
H11	0.9154	0.1203	0.2604	0.077*
C12	0.97669 (19)	0.3746 (9)	0.3345 (3)	0.0728 (13)
H12	1.0192	0.3065	0.3351	0.087*
C13	0.97758 (19)	0.5875 (8)	0.3786 (2)	0.0674 (12)
C14	0.91340 (19)	0.6792 (8)	0.3770 (2)	0.0700 (11)
H14	0.9122	0.8205	0.4063	0.084*
C15	0.85109 (19)	0.5671 (8)	0.3333 (2)	0.0629 (11)
H15	0.8087	0.6336	0.3339	0.075*
C16	1.04571 (19)	0.7126 (9)	0.4272 (3)	0.0934 (15)
H16A	1.0464	0.8690	0.4033	0.140*
H16B	1.0849	0.6210	0.4227	0.140*
H16C	1.0492	0.7273	0.4865	0.140*
C17	0.73954 (16)	0.5733 (7)	0.1260 (2)	0.0510 (10)
C18	0.79532 (18)	0.5398 (8)	0.0943 (2)	0.0677 (12)
H18	0.8257	0.4106	0.1129	0.081*
C19	0.80556 (19)	0.6973 (9)	0.0355 (2)	0.0763 (13)
H19	0.8439	0.6755	0.0162	0.092*
C20	0.76105 (19)	0.8849 (9)	0.0048 (2)	0.0723 (12)
H20	0.7683	0.9895	−0.0354	0.087*
C21	0.70498 (17)	0.9152 (8)	0.0348 (2)	0.0686 (12)
H21	0.6739	1.0415	0.0140	0.082*
C22	0.69399 (16)	0.7640 (7)	0.0945 (2)	0.0572 (11)
H22	0.6559	0.7888	0.1140	0.069*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0643 (13)	0.129 (2)	0.1097 (19)	−0.0038 (14)	0.0529 (13)	0.0221 (16)
N1	0.0469 (15)	0.075 (2)	0.0486 (17)	0.0069 (16)	0.0208 (14)	0.0043 (17)
N2	0.0556 (16)	0.082 (2)	0.0600 (18)	0.0223 (17)	0.0312 (15)	0.0262 (18)
C1	0.0478 (19)	0.073 (3)	0.051 (2)	0.000 (2)	0.0143 (18)	0.008 (2)
C2	0.0445 (19)	0.080 (3)	0.060 (2)	0.004 (2)	0.0172 (18)	0.002 (2)
C3	0.0378 (18)	0.100 (4)	0.058 (2)	−0.014 (2)	0.0207 (18)	0.004 (2)
C4	0.053 (2)	0.084 (3)	0.065 (2)	−0.013 (2)	0.023 (2)	0.014 (2)
C5	0.0502 (19)	0.066 (3)	0.058 (2)	−0.006 (2)	0.0187 (18)	0.005 (2)

C6	0.0395 (17)	0.068 (3)	0.0424 (18)	−0.0103 (19)	0.0103 (15)	−0.003 (2)
C7	0.0457 (18)	0.066 (3)	0.0413 (18)	0.0001 (19)	0.0157 (16)	0.002 (2)
C8	0.059 (2)	0.068 (3)	0.063 (2)	0.004 (2)	0.0263 (18)	0.005 (2)
C9	0.056 (2)	0.065 (3)	0.055 (2)	0.012 (2)	0.0271 (18)	0.011 (2)
C10	0.053 (2)	0.065 (3)	0.052 (2)	0.010 (2)	0.0276 (18)	0.011 (2)
C11	0.061 (2)	0.069 (3)	0.073 (3)	0.010 (2)	0.035 (2)	0.003 (2)
C12	0.057 (2)	0.087 (4)	0.083 (3)	0.012 (2)	0.035 (2)	0.020 (3)
C13	0.066 (2)	0.081 (3)	0.062 (2)	−0.004 (3)	0.031 (2)	0.015 (2)
C14	0.074 (3)	0.073 (3)	0.071 (3)	−0.004 (2)	0.035 (2)	−0.002 (2)
C15	0.061 (2)	0.069 (3)	0.067 (2)	0.011 (2)	0.031 (2)	0.004 (2)
C16	0.077 (3)	0.111 (4)	0.093 (3)	−0.022 (3)	0.028 (2)	0.014 (3)
C17	0.0434 (18)	0.071 (3)	0.0363 (18)	0.0020 (19)	0.0091 (16)	0.0070 (19)
C18	0.064 (2)	0.090 (3)	0.057 (2)	0.014 (2)	0.030 (2)	0.017 (2)
C19	0.058 (2)	0.119 (4)	0.057 (2)	0.007 (3)	0.026 (2)	0.015 (3)
C20	0.062 (2)	0.103 (4)	0.053 (2)	−0.006 (3)	0.019 (2)	0.022 (2)
C21	0.056 (2)	0.084 (3)	0.059 (2)	0.002 (2)	0.0089 (19)	0.020 (2)
C22	0.0396 (18)	0.080 (3)	0.048 (2)	0.001 (2)	0.0085 (16)	0.009 (2)

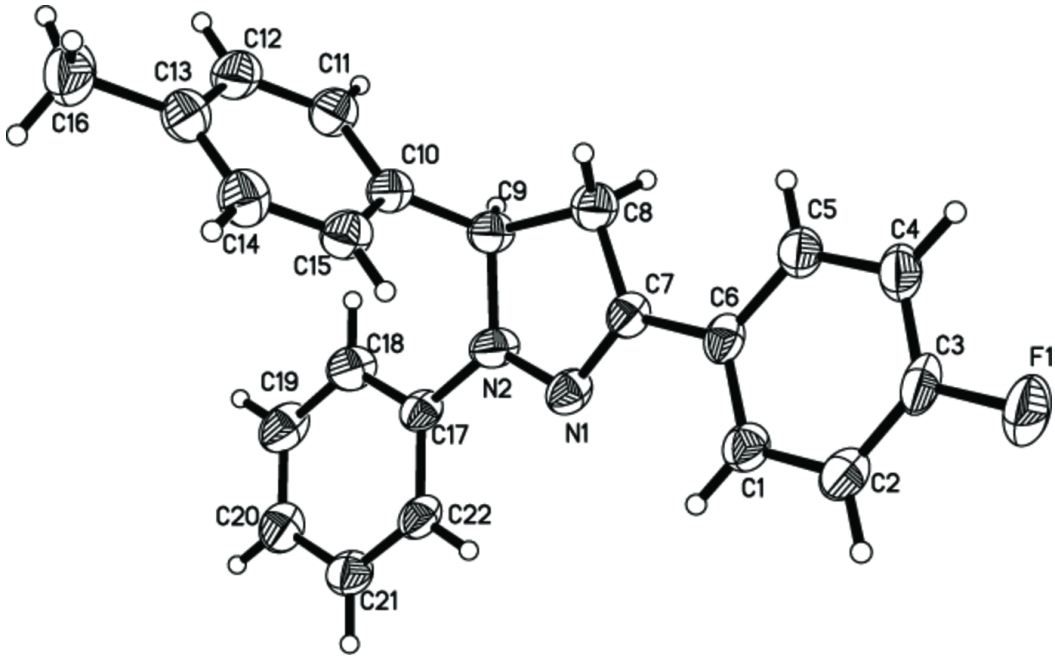
Geometric parameters (Å, °)

F1—C3	1.358 (4)	C11—C12	1.384 (5)
N1—C7	1.296 (4)	C11—H11	0.9300
N1—N2	1.382 (3)	C12—C13	1.392 (6)
N2—C17	1.388 (4)	C12—H12	0.9300
N2—C9	1.481 (4)	C13—C14	1.377 (5)
C1—C2	1.378 (4)	C13—C16	1.512 (5)
C1—C6	1.381 (5)	C14—C15	1.375 (5)
C1—H1	0.9300	C14—H14	0.9300
C2—C3	1.363 (5)	C15—H15	0.9300
C2—H2	0.9300	C16—H16A	0.9600
C3—C4	1.356 (5)	C16—H16B	0.9600
C4—C5	1.384 (4)	C16—H16C	0.9600
C4—H4	0.9300	C17—C22	1.390 (5)
C5—C6	1.392 (5)	C17—C18	1.392 (4)
C5—H5	0.9300	C18—C19	1.373 (5)
C6—C7	1.459 (4)	C18—H18	0.9300
C7—C8	1.503 (5)	C19—C20	1.363 (6)
C8—C9	1.538 (4)	C19—H19	0.9300
C8—H8A	0.9700	C20—C21	1.377 (5)
C8—H8B	0.9700	C20—H20	0.9300
C9—C10	1.508 (5)	C21—C22	1.368 (5)
C9—H9	0.9800	C21—H21	0.9300
C10—C11	1.380 (5)	C22—H22	0.9300
C10—C15	1.395 (5)		
C7—N1—N2	109.2 (3)	C10—C11—C12	120.9 (4)
N1—N2—C17	119.5 (3)	C10—C11—H11	119.5
N1—N2—C9	112.3 (3)	C12—C11—H11	119.5
C17—N2—C9	126.5 (3)	C11—C12—C13	121.7 (4)
C2—C1—C6	121.4 (3)	C11—C12—H12	119.2

C2—C1—H1	119.3	C13—C12—H12	119.2
C6—C1—H1	119.3	C14—C13—C12	117.0 (4)
C3—C2—C1	118.3 (3)	C14—C13—C16	121.1 (4)
C3—C2—H2	120.9	C12—C13—C16	121.9 (4)
C1—C2—H2	120.9	C15—C14—C13	121.7 (4)
C4—C3—F1	119.0 (4)	C15—C14—H14	119.2
C4—C3—C2	122.9 (3)	C13—C14—H14	119.2
F1—C3—C2	118.1 (4)	C14—C15—C10	121.4 (3)
C3—C4—C5	118.4 (4)	C14—C15—H15	119.3
C3—C4—H4	120.8	C10—C15—H15	119.3
C5—C4—H4	120.8	C13—C16—H16A	109.5
C4—C5—C6	120.9 (4)	C13—C16—H16B	109.5
C4—C5—H5	119.6	H16A—C16—H16B	109.5
C6—C5—H5	119.6	C13—C16—H16C	109.5
C1—C6—C5	118.2 (3)	H16A—C16—H16C	109.5
C1—C6—C7	121.6 (3)	H16B—C16—H16C	109.5
C5—C6—C7	120.3 (3)	N2—C17—C22	121.7 (3)
N1—C7—C6	121.1 (3)	N2—C17—C18	120.0 (3)
N1—C7—C8	112.6 (3)	C22—C17—C18	118.2 (3)
C6—C7—C8	126.3 (3)	C19—C18—C17	120.1 (4)
C7—C8—C9	103.1 (3)	C19—C18—H18	120.0
C7—C8—H8A	111.1	C17—C18—H18	120.0
C9—C8—H8A	111.1	C20—C19—C18	121.7 (3)
C7—C8—H8B	111.1	C20—C19—H19	119.1
C9—C8—H8B	111.1	C18—C19—H19	119.1
H8A—C8—H8B	109.1	C19—C20—C21	118.3 (3)
N2—C9—C10	111.9 (3)	C19—C20—H20	120.9
N2—C9—C8	100.9 (2)	C21—C20—H20	120.9
C10—C9—C8	115.0 (3)	C22—C21—C20	121.5 (4)
N2—C9—H9	109.6	C22—C21—H21	119.2
C10—C9—H9	109.6	C20—C21—H21	119.2
C8—C9—H9	109.6	C21—C22—C17	120.2 (3)
C11—C10—C15	117.3 (3)	C21—C22—H22	119.9
C11—C10—C9	121.7 (3)	C17—C22—H22	119.9
C15—C10—C9	121.0 (3)		
C7—N1—N2—C17	−175.0 (3)	N2—C9—C10—C11	−133.7 (3)
C7—N1—N2—C9	−8.9 (4)	C8—C9—C10—C11	112.0 (4)
C6—C1—C2—C3	0.9 (5)	N2—C9—C10—C15	44.9 (4)
C1—C2—C3—C4	−1.2 (6)	C8—C9—C10—C15	−69.4 (4)
C1—C2—C3—F1	179.5 (3)	C15—C10—C11—C12	−0.4 (5)
F1—C3—C4—C5	−179.7 (3)	C9—C10—C11—C12	178.3 (3)
C2—C3—C4—C5	1.1 (6)	C10—C11—C12—C13	−0.6 (6)
C3—C4—C5—C6	−0.6 (5)	C11—C12—C13—C14	1.1 (6)
C2—C1—C6—C5	−0.4 (5)	C11—C12—C13—C16	−179.6 (3)
C2—C1—C6—C7	179.9 (3)	C12—C13—C14—C15	−0.6 (6)
C4—C5—C6—C1	0.3 (5)	C16—C13—C14—C15	−179.9 (3)
C4—C5—C6—C7	180.0 (3)	C13—C14—C15—C10	−0.5 (6)
N2—N1—C7—C6	−178.4 (3)	C11—C10—C15—C14	1.0 (5)
N2—N1—C7—C8	−0.5 (4)	C9—C10—C15—C14	−177.7 (3)

C1—C6—C7—N1	−7.8 (5)	N1—N2—C17—C22	−2.5 (5)
C5—C6—C7—N1	172.5 (3)	C9—N2—C17—C22	−166.4 (3)
C1—C6—C7—C8	174.6 (3)	N1—N2—C17—C18	179.0 (3)
C5—C6—C7—C8	−5.1 (5)	C9—N2—C17—C18	15.1 (5)
N1—C7—C8—C9	8.9 (4)	N2—C17—C18—C19	−179.4 (4)
C6—C7—C8—C9	−173.3 (3)	C22—C17—C18—C19	2.1 (5)
N1—N2—C9—C10	−109.1 (3)	C17—C18—C19—C20	−1.9 (6)
C17—N2—C9—C10	55.8 (4)	C18—C19—C20—C21	0.5 (6)
N1—N2—C9—C8	13.7 (4)	C19—C20—C21—C22	0.6 (6)
C17—N2—C9—C8	178.6 (3)	C20—C21—C22—C17	−0.4 (6)
C7—C8—C9—N2	−12.6 (3)	N2—C17—C22—C21	−179.5 (3)
C7—C8—C9—C10	108.0 (3)	C18—C17—C22—C21	−0.9 (5)

Fig. 1


C12
C11
C13
C16
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C4
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C15
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C18
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F1
C19
N2
N1
C17
C1
C2
C20
C22
C21