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(*E*)-5-Phenyl-*N*′-(1-phenylethylidene)-1*H*-pyrazole-3-carbohydrazide

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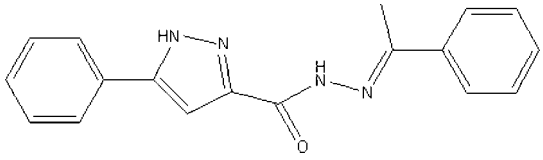
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; R factor = 0.054; wR factor = 0.143; data-to-parameter ratio = 8.1.

In the molecule of the title compound, $C_{18}H_{16}N_4O$, the intramolecular N—H···N hydrogen bond results in the formation of a planar five-membered ring, which is also coplanar with the adjacent five-membered ring, being oriented at a dihedral angle of 1.23 (3)°. The dihedral angles formed by the planar pyrazole ring with the adjacent phenyl ring and the other phenyl ring are 7.29 and 11.21°, respectively. The dihedral angle between the two phenyl rings is 18.07°. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules.

Related literature

For general background, see: Ogretir *et al.* (2006); Tarafder *et al.* (2000); Deschamps *et al.* (2003); Wu *et al.* (2006). For related literature, see: Yang & Raptis (2003); Ali *et al.* (2005). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{18}H_{16}N_4O$
M_r = 304.35
Tetragonal, $P4_3$
a = 8.0190 (11) Å
c = 24.147 (5) Å
V = 1552.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 294 (2) K
0.25 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
3737 measured reflections
1686 independent reflections
1017 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
3 standard reflections
every 100 reflections
intensity decay: 4.1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.143
S = 1.04
1686 reflections
209 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 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
N2—H2A···N3	0.86	2.34	2.714 (2)	107
N4—H4A···O1^{i}	0.86	1.98	2.788 (2)	157

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Siemens, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2398).

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(*E*)-5-Phenyl-*N*′-(1-phenylethylidene)-1*H*-pyrazole-3-carbohydrazide

Yongqi Qin, Fangfang Jian, Hailian Xiao and Jing Zhang

S1. Comment

Schiff bases have been used extensively as ligands in the field of coordination chemistry (Ogretir *et al.*, 2006). As dinegatively charged ligands, Schiff bases show potential as antimicrobial and anticancer agents (Tarafder *et al.*, 2000; Deschamps *et al.*, 2003) and so have biochemical and pharmacological applications. In addition, the chemical behavior of metal complexes with Schiff base ligands has attracted much attention because of their catalytic activity in some industrial and biochemical processes (Wu *et al.*, 2006). The title compound, (I), was synthesized as part of our study of these ligands and we report herein its crystal structure.

In the molecule of (I), (Fig. 1) the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). They are in good agreement with the corresponding values reported (Yang & Raptis, 2003). The C7—N1 [1.285 (6) Å] bond has a double-bond character (Ali *et al.*, 2005). The intramolecular N—H···N hydrogen bond (Table 1) results in the formation of a planar five-membered ring B (N2/H2A/N3/C9/C1). Rings A (C1—C6), C (N3/N4/C10—C11) and D (C13—C18) are, of course, planar and the dihedral angles between them are A/B = 11.06 (3)°, A/C = 11.10 (3)°, A/D = 17.82 (2)°, B/C = 1.23 (3)°, B/D = 7.85 (3)° and C/D = 7.15 (3)°. So, rings B and C are also co-planar.

In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 1) link the molecules, in which they may be effective in the stabilization of the structure.

S2. Experimental

A mixture of 5-phenyl-1*H*-pyrazole-3-carbohydrazide (10 mmol) with acetophenone (10 mmol) was stirred in refluxing ethanol (30 ml) for 5 h to afford the title compound, (I), (yield; 81%). Single crystals suitable for X-ray analysis were obtained by recrystallization from dimethylformamide (DMF) at 309 K.

S3. Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*N*), where x = 1.5 for methyl H, and x = 1.2 for all other H atoms.

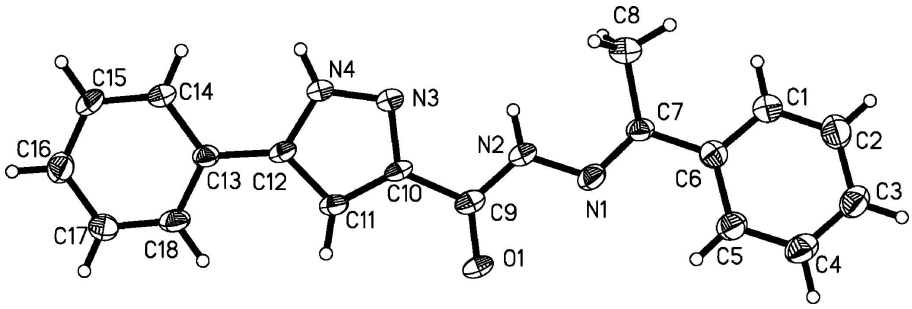

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

(*E*)-5-Phenyl-*N*′-(1-phenylethylidene)-1*H*-pyrazole-3-carbohydrazide

Crystal data

$C_{18}H_{16}N_4O$
$M_r = 304.35$
Tetragonal, $P4_3$
Hall symbol: P 4cw
$a = 8.0190\ (11)$ Å
$c = 24.147\ (5)$ Å
$V = 1552.8\ (4)$ Å^3
$Z = 4$
$F(000) = 640$
$D_x = 1.302$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 1$–$25°$
$\mu = 0.08$ mm^{-1}
$T = 294$ K
Block, yellow
0.25 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
3737 measured reflections
1686 independent reflections
1017 reflections with $I > 2\sigma(I)$
$R_{int} = 0.081$
$\theta_{max} = 26.9°$, $\theta_{min} = 2.5°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 9$
$l = -28 \rightarrow 28$
3 standard reflections every 100 reflections
intensity decay: 4.1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.143$
$S = 1.04$
1686 reflections
209 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0792P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ 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}
O1	0.6526 (4)	0.7885 (4)	0.19156 (14)	0.0530 (9)
N1	0.8996 (5)	0.5984 (5)	0.14452 (17)	0.0484 (9)
N2	0.7574 (4)	0.6473 (5)	0.11695 (18)	0.0495 (10)
H2A	0.7423	0.6186	0.0830	0.059*
N3	0.4872 (4)	0.7339 (5)	0.05571 (16)	0.0477 (10)
N4	0.3421 (5)	0.7929 (5)	0.03787 (17)	0.0467 (9)
H4A	0.3060	0.7777	0.0047	0.056*
C1	1.2768 (6)	0.3604 (6)	0.1231 (3)	0.0600 (14)
H1B	1.2546	0.3129	0.0888	0.072*
C2	1.4206 (7)	0.3174 (7)	0.1507 (3)	0.0700 (16)
H2B	1.4918	0.2381	0.1355	0.084*
C3	1.4605 (6)	0.3911 (7)	0.2008 (3)	0.0615 (14)
H3B	1.5600	0.3658	0.2187	0.074*
C4	1.3495 (7)	0.5025 (7)	0.2236 (2)	0.0624 (14)
H4B	1.3737	0.5505	0.2578	0.075*
C5	1.2040 (6)	0.5445 (6)	0.1972 (2)	0.0547 (13)
H5A	1.1314	0.6203	0.2135	0.066*
C6	1.1639 (5)	0.4735 (6)	0.1456 (2)	0.0445 (11)
C7	1.0116 (6)	0.5222 (5)	0.1161 (2)	0.0427 (10)
C8	0.9950 (7)	0.4862 (7)	0.0543 (2)	0.0612 (14)
H8A	0.9620	0.5861	0.0354	0.092*
H8B	0.9123	0.4013	0.0487	0.092*
H8C	1.1002	0.4485	0.0401	0.092*
C9	0.6421 (6)	0.7398 (5)	0.1432 (2)	0.0445 (11)
C10	0.4972 (5)	0.7848 (5)	0.10870 (19)	0.0401 (9)
C11	0.3586 (5)	0.8767 (5)	0.12334 (19)	0.0424 (10)
H11A	0.3378	0.9267	0.1574	0.051*
C12	0.2562 (5)	0.8794 (5)	0.07682 (19)	0.0403 (10)
C13	0.0915 (5)	0.9501 (5)	0.06862 (18)	0.0384 (10)
C14	0.0112 (6)	0.9482 (7)	0.0169 (2)	0.0552 (13)
H14A	0.0658	0.9053	−0.0140	0.066*
C15	−0.1487 (7)	1.0101 (7)	0.0119 (3)	0.0645 (15)
H15A	−0.2025	1.0052	−0.0221	0.077*
C16	−0.2297 (6)	1.0789 (7)	0.0566 (2)	0.0594 (14)
H16A	−0.3361	1.1233	0.0525	0.071*

C17	−0.1517 (6)	1.0818 (7)	0.1080 (2)	0.0581 (13)
H17A	−0.2059	1.1264	0.1386	0.070*
C18	0.0066 (6)	1.0182 (6)	0.1129 (2)	0.0514 (12)
H18A	0.0584	1.0211	0.1474	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.064 (2)	0.071 (2)	0.024 (2)	0.0045 (16)	−0.0093 (14)	−0.0017 (15)
N1	0.052 (2)	0.058 (2)	0.035 (3)	0.0014 (18)	−0.0090 (18)	0.0042 (18)
N2	0.052 (2)	0.066 (2)	0.030 (2)	0.0052 (19)	−0.0083 (18)	−0.0035 (19)
N3	0.048 (2)	0.062 (2)	0.033 (2)	0.0054 (18)	−0.0069 (17)	−0.0105 (17)
N4	0.050 (2)	0.061 (2)	0.029 (2)	0.0088 (18)	−0.0065 (17)	−0.0093 (17)
C1	0.056 (3)	0.071 (3)	0.053 (4)	0.002 (3)	−0.003 (3)	−0.012 (3)
C2	0.056 (3)	0.072 (4)	0.082 (5)	0.005 (3)	0.000 (3)	−0.011 (3)
C3	0.055 (3)	0.075 (3)	0.054 (4)	−0.003 (3)	−0.010 (3)	0.009 (3)
C4	0.067 (3)	0.075 (3)	0.044 (4)	−0.008 (3)	−0.008 (3)	0.004 (3)
C5	0.060 (3)	0.056 (3)	0.048 (4)	0.002 (2)	−0.007 (2)	−0.002 (2)
C6	0.045 (2)	0.045 (2)	0.043 (3)	−0.005 (2)	0.004 (2)	0.005 (2)
C7	0.047 (2)	0.052 (2)	0.029 (3)	−0.004 (2)	0.0008 (19)	−0.001 (2)
C8	0.067 (3)	0.076 (3)	0.041 (4)	0.004 (3)	0.001 (2)	−0.004 (2)
C9	0.053 (3)	0.050 (3)	0.031 (3)	−0.009 (2)	−0.004 (2)	0.003 (2)
C10	0.049 (2)	0.050 (2)	0.022 (2)	−0.003 (2)	−0.0065 (18)	−0.0004 (18)
C11	0.055 (3)	0.046 (2)	0.027 (3)	−0.001 (2)	−0.003 (2)	−0.0047 (18)
C12	0.049 (2)	0.047 (2)	0.024 (3)	−0.0011 (19)	−0.0063 (18)	−0.0003 (19)
C13	0.047 (2)	0.042 (2)	0.026 (3)	0.0015 (18)	0.0018 (18)	0.0020 (18)
C14	0.064 (3)	0.074 (3)	0.028 (3)	0.018 (3)	−0.005 (2)	−0.003 (2)
C15	0.068 (3)	0.095 (4)	0.031 (3)	0.022 (3)	−0.011 (2)	0.007 (3)
C16	0.054 (3)	0.075 (3)	0.049 (4)	0.015 (2)	0.001 (2)	0.012 (2)
C17	0.059 (3)	0.077 (3)	0.039 (3)	0.013 (3)	0.009 (2)	0.000 (2)
C18	0.060 (3)	0.068 (3)	0.026 (3)	0.000 (2)	0.002 (2)	−0.002 (2)

Geometric parameters (Å, º)

O1—C9	1.234 (6)	C7—C8	1.526 (7)
N1—C7	1.285 (6)	C8—H8A	0.9600
N1—N2	1.378 (5)	C8—H8B	0.9600
N2—C9	1.344 (6)	C8—H8C	0.9600
N2—H2A	0.8600	C9—C10	1.474 (6)
N3—N4	1.328 (5)	C10—C11	1.380 (6)
N3—C10	1.346 (6)	C11—C12	1.392 (6)
N4—C12	1.357 (6)	C11—H11A	0.9300
N4—H4A	0.8600	C12—C13	1.451 (6)
C1—C2	1.375 (8)	C13—C18	1.381 (7)
C1—C6	1.391 (7)	C13—C14	1.406 (7)
C1—H1B	0.9300	C14—C15	1.380 (7)
C2—C3	1.385 (8)	C14—H14A	0.9300
C2—H2B	0.9300	C15—C16	1.376 (8)

C3—C4	1.376 (8)	C15—H15A	0.9300
C3—H3B	0.9300	C16—C17	1.389 (7)
C4—C5	1.371 (7)	C16—H16A	0.9300
C4—H4B	0.9300	C17—C18	1.373 (7)
C5—C6	1.408 (7)	C17—H17A	0.9300
C5—H5A	0.9300	C18—H18A	0.9300
C6—C7	1.466 (7)		

C7—N1—N2	117.2 (4)	H8A—C8—H8C	109.5
C9—N2—N1	119.9 (4)	H8B—C8—H8C	109.5
C9—N2—H2A	120.1	O1—C9—N2	125.0 (4)
N1—N2—H2A	120.1	O1—C9—C10	120.8 (4)
N4—N3—C10	104.6 (4)	N2—C9—C10	114.2 (4)
N3—N4—C12	113.7 (4)	N3—C10—C11	110.9 (4)
N3—N4—H4A	123.1	N3—C10—C9	120.7 (4)
C12—N4—H4A	123.1	C11—C10—C9	128.4 (4)
C2—C1—C6	121.4 (6)	C10—C11—C12	106.1 (4)
C2—C1—H1B	119.3	C10—C11—H11A	127.0
C6—C1—H1B	119.3	C12—C11—H11A	127.0
C1—C2—C3	120.6 (5)	N4—C12—C11	104.6 (4)
C1—C2—H2B	119.7	N4—C12—C13	124.6 (4)
C3—C2—H2B	119.7	C11—C12—C13	130.8 (4)
C4—C3—C2	118.5 (5)	C18—C13—C14	117.9 (4)
C4—C3—H3B	120.7	C18—C13—C12	119.8 (4)
C2—C3—H3B	120.7	C14—C13—C12	122.3 (4)
C5—C4—C3	121.6 (5)	C15—C14—C13	119.9 (5)
C5—C4—H4B	119.2	C15—C14—H14A	120.1
C3—C4—H4B	119.2	C13—C14—H14A	120.1
C4—C5—C6	120.5 (5)	C16—C15—C14	121.0 (5)
C4—C5—H5A	119.8	C16—C15—H15A	119.5
C6—C5—H5A	119.8	C14—C15—H15A	119.5
C1—C6—C5	117.4 (4)	C15—C16—C17	119.6 (5)
C1—C6—C7	121.8 (5)	C15—C16—H16A	120.2
C5—C6—C7	120.8 (4)	C17—C16—H16A	120.2
N1—C7—C6	116.8 (4)	C18—C17—C16	119.2 (5)
N1—C7—C8	123.4 (4)	C18—C17—H17A	120.4
C6—C7—C8	119.8 (4)	C16—C17—H17A	120.4
C7—C8—H8A	109.5	C17—C18—C13	122.3 (5)
C7—C8—H8B	109.5	C17—C18—H18A	118.8
H8A—C8—H8B	109.5	C13—C18—H18A	118.8
C7—C8—H8C	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···N3	0.86	2.34	2.714 (2)	107

N4—H4*A*···O1^{i}	0.86	1.98	2.788 (2)	157

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