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3-Amino-1-phenyl-4-(propan-2-ylidene)-pyrazol-5(4*H*)-one

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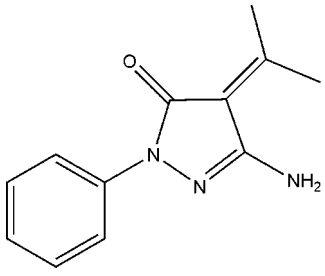
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.005 Å; R factor = 0.049; wR factor = 0.138; data-to-parameter ratio = 9.9.

In the title molecule, $C_{12}H_{13}N_3O$, the phenyl and the pyrazole rings make a dihedral angle of 7.5 (2)°. Intermolecular N—H···O hydrogen bonds involving the amino group link the molecules into a three-dimensional framework.

Related literature

For a related structure, see: Wang *et al.* (2003). For applications of pyrazolone derivatives, see: Hodnett *et al.* (1972).



Experimental

Crystal data

$C_{12}H_{13}N_3O$
M_r = 215.25
Orthorhombic, *Fdd*2
a = 22.557 (8) Å
b = 26.291 (9) Å
c = 7.528 (3) Å
V = 4465 (3) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 273 (2) K
0.15 × 0.12 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.987, T_{max} = 0.993
7064 measured reflections
1448 independent reflections
960 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.138
S = 1.03
1448 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N2^{i}	0.86	2.25	3.105 (3)	174
N3—H3B···O1^{ii}	0.86	2.32	3.054 (3)	144
C5—H5A···O1	0.96	2.20	2.935 (5)	131
C12—H12···O1	0.93	2.26	2.882 (4)	124

Symmetry codes: (i) $-x+1, -y, z$; (ii) $-x+\frac{3}{4}, y-\frac{1}{4}, z+\frac{1}{4}$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: CI2526).

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supporting information

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3-Amino-1-phenyl-4-(propan-2-ylidene)pyrazol-5(4*H*)-one

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S1. Comment

Pyrazolone derivatives are well known for their applications as analgesics, antipyretics, anti-inflammatory and insecticides (Hodnett & Paul, 1972). Therefore, the study on the derivatives of pyrazolone is the focus of many research groups working in the fields of coordination chemistry, biomedicine and pharmaceutical chemistry. We report here the crystal structure of the title compound.

All geometric parameters in the title molecule (Fig. 1) are in good agreement with those found in *N*-(1,5-dihydro-1-phenyl-3-methyl-4-benzoyl)-3-chloroaniline (Wang *et al.*, 2003). The benzene and the pyrazole rings make a dihedral angle of 7.5 (2)°. Intermolecular N—H···O hydrogen bonds involving the amino group link the molecules into a three-dimensional framework (Fig. 2).

S2. Experimental

3-Amino-1-phenyl-5-pyrazolone (0.175 g, 1 mmol) was added to acetone (20 ml), and the mixture was stirred under reflux at 343 K for 6 h. The solution was allowed to cool to room temperature and filtered. Orange crystals suitable for X-ray diffraction study were obtained after 7 d (yield 0.172 g, 80%; m.p. 370–372 K). Analysis found: C 66.90, H 7.02, N 19.48%; $C_{12}H_{13}N_3O$ requires: C 66.96, H 6.09, N 19.52%.

S3. Refinement

H atoms were positioned geometrically (C—H = 0.93 - 0.96 Å and N—H = 0.86 Å) and refined as riding, with U_{iso}(H) = 1.5U_{eq}(C_{methyl}) and U_{iso}(H) = 1.2U_{eq}(N and $C_{aromatic}$). In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

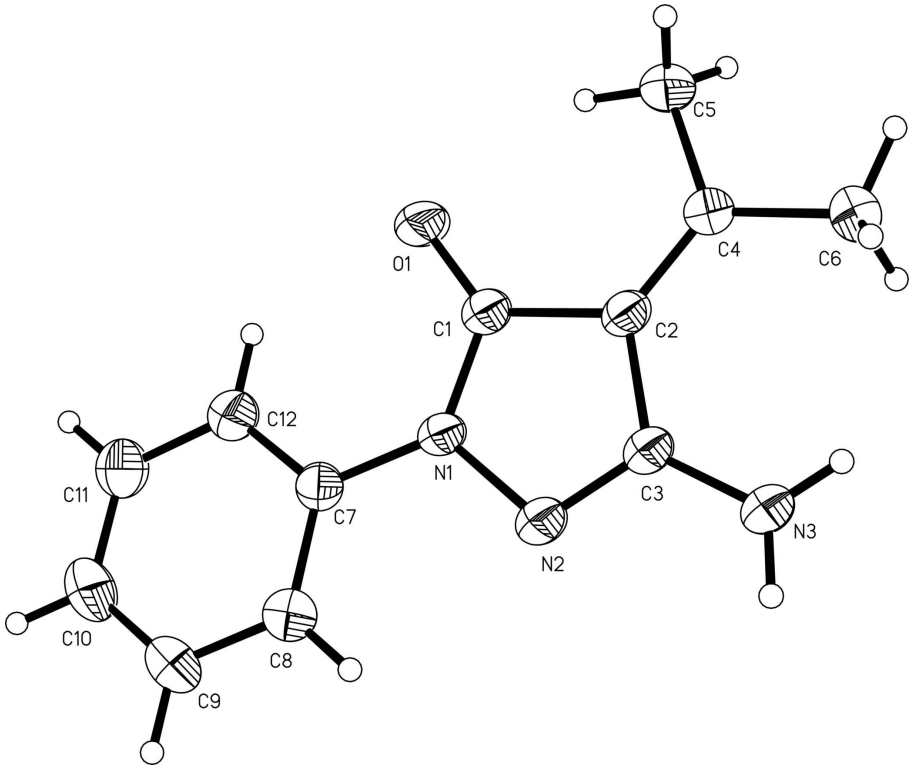

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

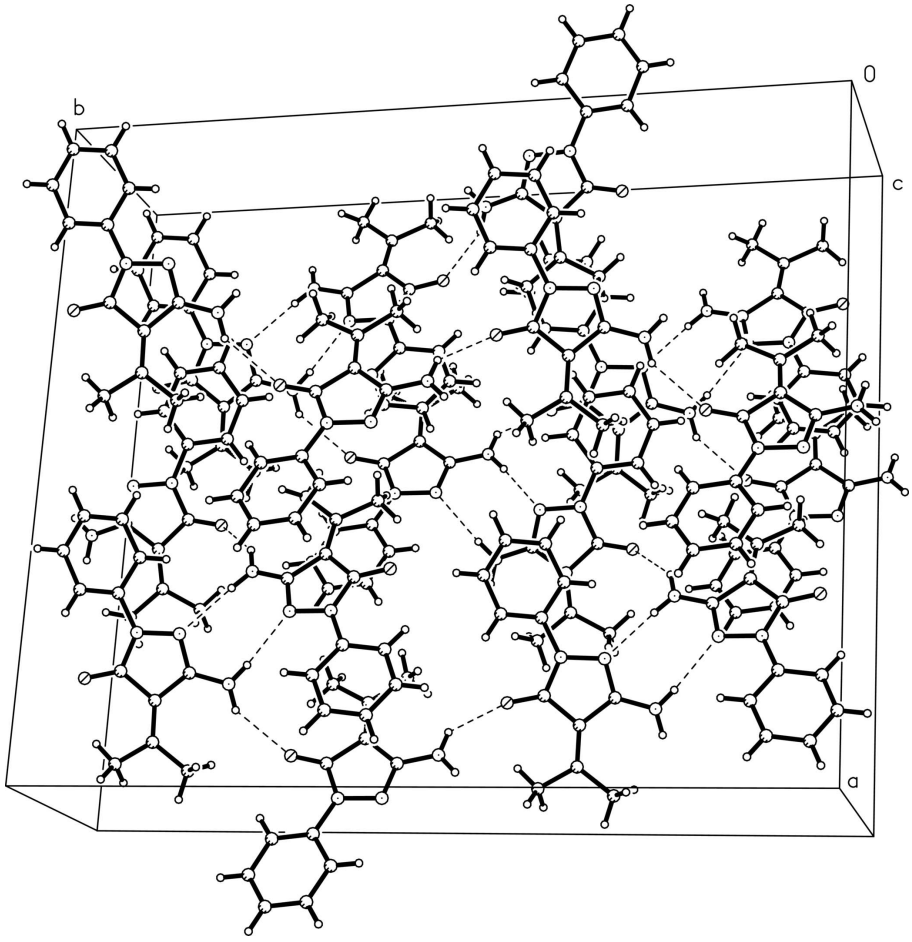

Figure 2

Crystal packing of the title compound.

3-Amino-1-phenyl-4-(propan-2-ylidene)pyrazol-5(4*H*)-one

Crystal data

$C_{12}H_{13}N_3O$
$M_r = 215.25$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 22.557\ (8)$ Å
$b = 26.291\ (9)$ Å
$c = 7.528\ (3)$ Å
$V = 4465\ (3)$ Å^3
$Z = 16$
$F(000) = 1824$
$D_x = 1.281$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1182 reflections
$\theta = 3.0$–$21.1°$
$\mu = 0.09$ mm^{-1}
$T = 273$ K
Block, orange
0.15 × 0.12 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.987$, $T_{max} = 0.993$
7064 measured reflections
1448 independent reflections
960 reflections with $I > 2\sigma(I)$

R_{int} = 0.049
θ_{max} = 28.3°, θ_{min} = 2.4°
h = −30→30
k = −35→35
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.138
S = 1.03
1448 reflections
147 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 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	0.42250 (10)	0.19174 (8)	0.4851 (4)	0.0682 (8)
N1	0.48595 (11)	0.12402 (8)	0.4474 (4)	0.0491 (7)
N2	0.48444 (12)	0.07007 (8)	0.4631 (4)	0.0527 (7)
N3	0.41722 (12)	0.00919 (9)	0.5431 (5)	0.0677 (9)
H3A	0.4432	−0.0143	0.5262	0.081*
H3B	0.3821	0.0013	0.5779	0.081*
C1	0.43317 (14)	0.14594 (11)	0.4913 (5)	0.0493 (8)
C2	0.39362 (13)	0.10311 (10)	0.5399 (5)	0.0461 (8)
C3	0.43167 (14)	0.05839 (10)	0.5153 (5)	0.0484 (8)
C4	0.33718 (14)	0.10759 (11)	0.5946 (5)	0.0496 (8)
C5	0.30732 (16)	0.15770 (12)	0.6224 (6)	0.0622 (10)
H5A	0.3365	0.1842	0.6240	0.093*
H5B	0.2865	0.1573	0.7335	0.093*
H5C	0.2798	0.1637	0.5275	0.093*
C6	0.29806 (16)	0.06265 (13)	0.6325 (7)	0.0685 (12)
H6A	0.2930	0.0431	0.5260	0.103*
H6B	0.2601	0.0743	0.6733	0.103*
H6C	0.3161	0.0419	0.7224	0.103*
C7	0.53990 (13)	0.14704 (11)	0.3951 (5)	0.0471 (7)
C8	0.58725 (15)	0.11694 (14)	0.3435 (5)	0.0605 (9)
H8	0.5833	0.0817	0.3398	0.073*
C9	0.64021 (17)	0.13950 (15)	0.2978 (6)	0.0740 (12)

H9	0.6719	0.1192	0.2633	0.089*
C10	0.64711 (18)	0.19102 (16)	0.3021 (7)	0.0776 (12)
H10	0.6831	0.2058	0.2707	0.093*
C11	0.60048 (16)	0.22050 (14)	0.3530 (7)	0.0731 (12)
H11	0.6049	0.2557	0.3554	0.088*
C12	0.54684 (16)	0.19944 (13)	0.4011 (6)	0.0605 (10)
H12	0.5156	0.2201	0.4371	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0507 (13)	0.0357 (12)	0.118 (2)	0.0070 (9)	0.0051 (14)	0.0085 (12)
N1	0.0397 (14)	0.0350 (12)	0.0728 (19)	0.0038 (10)	−0.0001 (12)	0.0076 (13)
N2	0.0485 (15)	0.0354 (12)	0.074 (2)	0.0030 (11)	0.0015 (14)	0.0035 (13)
N3	0.0574 (17)	0.0351 (14)	0.111 (3)	0.0070 (12)	0.0143 (18)	0.0088 (15)
C1	0.0438 (17)	0.0390 (16)	0.065 (2)	0.0028 (12)	−0.0009 (16)	0.0060 (14)
C2	0.0464 (17)	0.0345 (14)	0.057 (2)	0.0027 (13)	−0.0045 (15)	0.0042 (14)
C3	0.0477 (18)	0.0341 (15)	0.063 (2)	0.0035 (12)	0.0000 (16)	0.0032 (14)
C4	0.0442 (18)	0.0455 (17)	0.059 (2)	0.0041 (13)	0.0001 (15)	0.0060 (14)
C5	0.053 (2)	0.053 (2)	0.081 (3)	0.0102 (15)	0.006 (2)	0.0052 (18)
C6	0.051 (2)	0.055 (2)	0.099 (3)	−0.0012 (15)	0.012 (2)	0.012 (2)
C7	0.0413 (17)	0.0493 (17)	0.0508 (19)	0.0001 (13)	−0.0011 (15)	0.0080 (16)
C8	0.056 (2)	0.0558 (19)	0.070 (3)	0.0046 (16)	0.0082 (19)	0.0041 (17)
C9	0.052 (2)	0.081 (3)	0.090 (3)	0.004 (2)	0.023 (2)	0.008 (2)
C10	0.053 (2)	0.082 (3)	0.098 (3)	−0.0153 (19)	0.016 (2)	0.009 (2)
C11	0.061 (2)	0.057 (2)	0.101 (3)	−0.0129 (17)	0.006 (2)	0.009 (2)
C12	0.052 (2)	0.0498 (19)	0.080 (3)	0.0001 (14)	0.0046 (18)	0.0082 (18)

Geometric parameters (Å, º)

O1—C1	1.229 (3)	C5—H5C	0.96
N1—C1	1.363 (4)	C6—H6A	0.96
N1—C7	1.415 (4)	C6—H6B	0.96
N1—N2	1.424 (3)	C6—H6C	0.96
N2—C3	1.290 (4)	C7—C8	1.385 (5)
N3—C3	1.350 (4)	C7—C12	1.387 (4)
N3—H3A	0.86	C8—C9	1.377 (5)
N3—H3B	0.86	C8—H8	0.93
C1—C2	1.482 (4)	C9—C10	1.364 (5)
C2—C4	1.343 (4)	C9—H9	0.93
C2—C3	1.468 (4)	C10—C11	1.362 (6)
C4—C5	1.494 (4)	C10—H10	0.93
C4—C6	1.502 (5)	C11—C12	1.379 (5)
C5—H5A	0.96	C11—H11	0.93
C5—H5B	0.96	C12—H12	0.93
C1—N1—C7	129.6 (2)	C4—C6—H6A	109.5
C1—N1—N2	112.3 (2)	C4—C6—H6B	109.5

C7—N1—N2	118.0 (2)	H6A—C6—H6B	109.5
C3—N2—N1	106.5 (2)	C4—C6—H6C	109.5
C3—N3—H3A	120.0	H6A—C6—H6C	109.5
C3—N3—H3B	120.0	H6B—C6—H6C	109.5
H3A—N3—H3B	120.0	C8—C7—C12	119.3 (3)
O1—C1—N1	125.2 (3)	C8—C7—N1	119.8 (3)
O1—C1—C2	129.5 (3)	C12—C7—N1	120.8 (3)
N1—C1—C2	105.3 (2)	C9—C8—C7	119.5 (3)
C4—C2—C3	131.6 (3)	C9—C8—H8	120.2
C4—C2—C1	125.4 (3)	C7—C8—H8	120.2
C3—C2—C1	103.0 (3)	C10—C9—C8	121.4 (3)
N2—C3—N3	119.9 (3)	C10—C9—H9	119.3
N2—C3—C2	112.8 (2)	C8—C9—H9	119.3
N3—C3—C2	127.3 (3)	C11—C10—C9	118.9 (3)
C2—C4—C5	123.2 (3)	C11—C10—H10	120.5
C2—C4—C6	123.1 (3)	C9—C10—H10	120.5
C5—C4—C6	113.7 (3)	C10—C11—C12	121.5 (4)
C4—C5—H5A	109.5	C10—C11—H11	119.2
C4—C5—H5B	109.5	C12—C11—H11	119.2
H5A—C5—H5B	109.5	C11—C12—C7	119.3 (3)
C4—C5—H5C	109.5	C11—C12—H12	120.4
H5A—C5—H5C	109.5	C7—C12—H12	120.4
H5B—C5—H5C	109.5		

C1—N1—N2—C3	0.2 (4)	C3—C2—C4—C5	175.8 (4)
C7—N1—N2—C3	178.7 (3)	C1—C2—C4—C5	−3.1 (5)
C7—N1—C1—O1	3.4 (6)	C3—C2—C4—C6	−4.6 (6)
N2—N1—C1—O1	−178.3 (3)	C1—C2—C4—C6	176.5 (4)
C7—N1—C1—C2	−178.3 (3)	C1—N1—C7—C8	−175.2 (4)
N2—N1—C1—C2	0.0 (4)	N2—N1—C7—C8	6.5 (5)
O1—C1—C2—C4	−2.8 (6)	C1—N1—C7—C12	7.3 (6)
N1—C1—C2—C4	179.0 (3)	N2—N1—C7—C12	−171.0 (3)
O1—C1—C2—C3	178.0 (4)	C12—C7—C8—C9	−0.5 (6)
N1—C1—C2—C3	−0.2 (4)	N1—C7—C8—C9	−178.1 (4)
N1—N2—C3—N3	−179.5 (3)	C7—C8—C9—C10	0.0 (7)
N1—N2—C3—C2	−0.3 (4)	C8—C9—C10—C11	0.1 (8)
C4—C2—C3—N2	−178.8 (4)	C9—C10—C11—C12	0.4 (8)
C1—C2—C3—N2	0.3 (4)	C10—C11—C12—C7	−0.9 (7)
C4—C2—C3—N3	0.3 (6)	C8—C7—C12—C11	1.0 (6)
C1—C2—C3—N3	179.4 (3)	N1—C7—C12—C11	178.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N2^{i}	0.86	2.25	3.105 (3)	174
N3—H3*B*···O1^{ii}	0.86	2.32	3.054 (3)	144

C5—H5*A*···O1	0.96	2.20	2.935 (5)	131
C12—H12···O1	0.93	2.26	2.882 (4)	124

Symmetry codes: (i) −*x*+1, −*y*, *z*; (ii) −*x*+3/4, *y*−1/4, *z*+1/4.