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5,5-Dimethyl-3-[(5-phenyl-1*H*-pyrazol-3-yl)amino]-cyclohex-2-en-1-one

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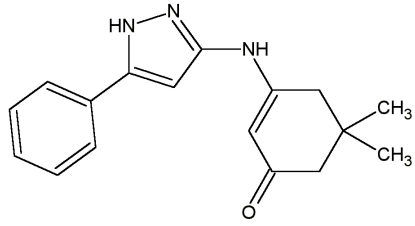
The title compound, $C_{17}H_{19}N_3O$, crystallizes with two molecules in the asymmetric unit. These molecules are hydrogen bonded intra- and intermolecularly *via* strong and weak hydrogen bonds to form a complex three-dimensional network.

Comment

The title compound, (I), was an intermediate in the preparation of pyrazolo[3,4-*b*]quinolin-5-ones. Geometric parameters are given in Table 1 and a view of each individual molecule is given in Figs. 1 and 2.



(I)

The primary supramolecular structure is a *C*(9) motif (Bernstein *et al.*, 1995) involving only molecule 1, N133—H133···O11^{ii} [symmetry code: (ii) $x, y-1, z$]. A similar chain perpendicular to the first is formed by molecule 2 and its symmetry-related molecules by the *C*(9) motif, N233—H233···O21^{iii} [symmetry code: (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, 1+z$]. These chains are then interlinked by further hydrogen bonds, N131—H131···O21^{i} and N231—H231···O11, which links the two molecules in the asymmetric unit [symmetry code: (i) $1-x, 1-y, z+\frac{1}{2}$]. These along with two weak C—H···N hydrogen bonds combine to form a complex three-dimensional network. The details of the hydrogen bonding are given in Table 2.

Examination of the structure with *PLATON* (Spek, 2000) showed that there were no solvent-accessible voids in the crystal lattice.

The high R_{int} value is due to slight interference from one or more small crystallites that were impossible to remove from the main crystal.

Experimental

A solution of 5-amino-3-phenylpyrazole (1 mmol) and dimedone (1 mmol) in 15 ml of absolute ethanol was heated to reflux for 25 min. The title compound appeared as a solid which was filtered off and washed with ethanol, dried and recrystallized from ethanol affording yellow crystals suitable for X-ray diffraction; yield 80%.

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.123
Data-to-parameter ratio = 9.4

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

Crystal data

$C_{17}H_{19}N_3O$
$M_r = 281.35$
Orthorhombic, $Pna2_1$
$a = 19.0427\ (2)$ Å
$b = 9.6024\ (4)$ Å
$c = 16.6135\ (7)$ Å
$V = 3037.87\ (18)$ Å^3
$Z = 8$
$D_x = 1.230$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2770 reflections
$\theta = 2.1$–$25.0°$
$\mu = 0.08$ mm^{-1}
$T = 150\ (1)$ K
Plate, colourless
$0.38 \times 0.18 \times 0.08$ mm

Data collection

KappaCCD diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.971$, $T_{max} = 0.994$
19 777 measured reflections
3597 independent reflections
2776 reflections with $I > 2\sigma(I)$
$R_{int} = 0.112$
$\theta_{max} = 27.5°$
$h = -24 \rightarrow 24$
$k = -11 \rightarrow 12$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.123$
$S = 0.99$
3597 reflections
383 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.010$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C13—N131	1.353 (4)	N231—C231	1.402 (4)
N131—C131	1.404 (4)	N232—C231	1.334 (4)
C131—N132	1.325 (4)	N232—N233	1.357 (3)
N132—N133	1.367 (4)	N233—C234	1.356 (4)
N133—C134	1.347 (4)		
C13—N131—C131	125.8 (2)	C23—N231—C231	128.3 (3)
C131—N132—N133	103.8 (2)	C231—N232—N233	103.8 (2)
C134—N133—N132	111.9 (2)	N232—N233—C234	112.7 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N131—H131···O21^{i}	0.88	2.02	2.881 (3)	167
N133—H133···O11^{ii}	0.88	1.93	2.788 (3)	164
N233—H233···O21^{iii}	0.88	1.98	2.825 (3)	162
N231—H231···O11	0.88	2.07	2.942 (4)	173
C237—H237···N133^{iv}	0.95	2.59	3.430 (4)	147
C240—H240···N133^{v}	0.95	2.57	3.479 (4)	160

Symmetry codes: (i) $1-x, 1-y, \frac{1}{2}+z$; (ii) $x, y-1, z$; (iii) $x-\frac{1}{2}, \frac{3}{2}-y, z$; (iv) $1-x, 1-y, z-\frac{1}{2}$; (v) $\frac{1}{2}-x, \frac{1}{2}+y, z-\frac{1}{2}$.

H atoms were treated as riding atoms with C—H distances in the range 0.95–0.99 Å and an N—H distance of 0.88 Å. No attempt was made to determine the absolute configuration since the structure contains only C, H, N and O atoms. Friedel pairs were merged.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

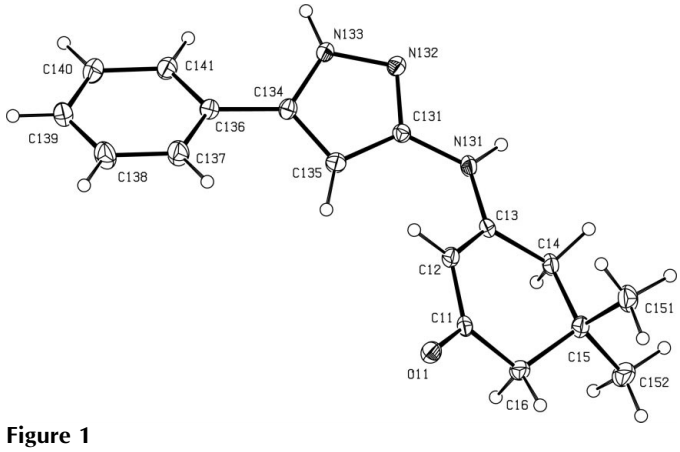


Figure 1
A view of molecule 1 with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

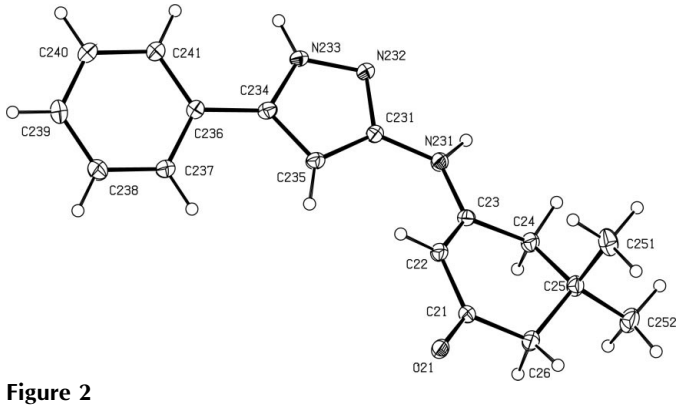


Figure 2
A view of molecule 2 with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

X-ray data were collected at the EPSRC, X-ray Crystallographic Service, University of Southampton, using an Enraf—Nonius KappaCCD diffractometer. The authors thank the staff for all their help and advice. We are grateful to the Ministerio de Educación y Cultura for the award of a grant to one of the authors (AQ).

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