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3,7,7-Trimethyl-1-phenyl-1,6,7,8-tetrahydro-5*H*-pyrazolo[3,4-*b*]quinolin-5-one

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.059
wR factor = 0.138
Data-to-parameter ratio = 16.8

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

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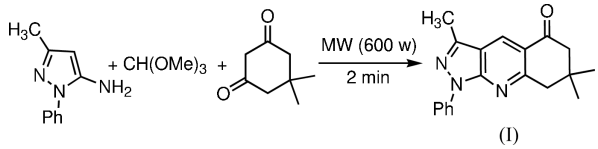
The title compound, $C_{19}H_{19}N_3O$, crystallizes in the space group $P\overline{1}$, with $Z' = 2$. Weak C—H···N, C—H···O and C—H···π interactions link the molecules into ribbons.

Comment

There are no unusual bond lengths or angles in the two molecules in the asymmetric unit of the title compound, (I), and there are no significant differences between corresponding bonds and bond angles of the two molecules. The ring-puckering parameters (Cremer & Pople, 1975) for the rings, C14*A*/C15/C16/C17/C18/C13*A* and C24*A*/C25/C26/C27/C28/C23*A*, show very little variation between molecules 1 and 2 (values for molecule 1 given first): Q = 0.491 (2) and 0.486 (2)°, θ = 127.1 (2) and 125.8 (3)°, and π = 311.0 (3) and 311.1 (3)°. However, the molecules differ in the torsion angles about the N11—C111 and N21—C211 bonds (Table 1, and Figs. 1 and 2).



(I)

There are no significant intermolecular interactions between molecules 1 and 2. There are two intramolecular C—H···O contacts, *viz*. C112···N19 in molecule 1 and C212···N29 in molecule 2 (Table 1). Weak C—H···O hydrogen bonds, C14···O15($1-x$, $2-y$, $1-z$) and C24···O25($-x$, $-y$, $-z$), link the molecules into dimers (Table 2), in each case forming $R_2^2(10)$ rings (Bernstein *et al.*, 1995). In addition, there are two weak C—H···π interactions, C16—H16*B*···*Cg*1($-x$, $2-y$, $1-z$) and C26—H16*B*···*Cg*2($1-x$, $-y$, $-z$); *Cg*1 is the centroid of ring C111–C116 and *Cg*2 is the centroid of ring C211–C216. These interactions link the dimers into ribbons of rings which run parallel to the *a* axis, and in the case of molecule 1, this ribbon is reinforced by a weak interaction between C113 and O17($x-1$, y, z), which forms a $C(11)$ chain from the molecules. In both cases, these ribbons of molecules run parallel to the *a* axis (Figs. 3 and 4).

Experimental

Equimolar amounts of 5-amino-3-methyl-1-phenylpyrazole, trimethyl orthoformate and dimedone were placed into Pyrex glass open vessels and irradiated in a domestic microwave oven for 2 min (at 600 W). The product of the reaction was recrystallized from absolute ethanol (yield 75%; m.p. 431 K). Analysis calculated for $C_{19}H_{19}N_3O$: C 74.73, H 6.27, N 13.76%; found: C 74.30, H 6.37, N 13.80%.

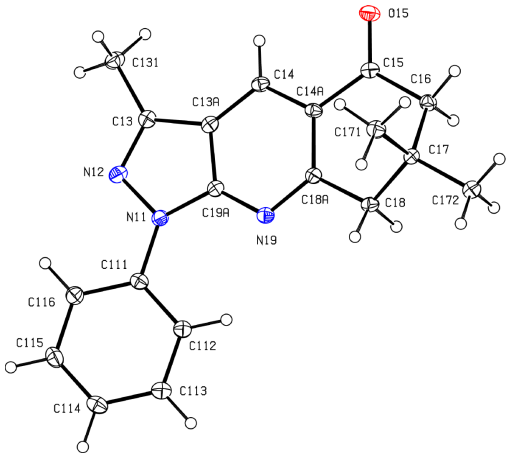


Figure 1
A view of molecule 1 of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

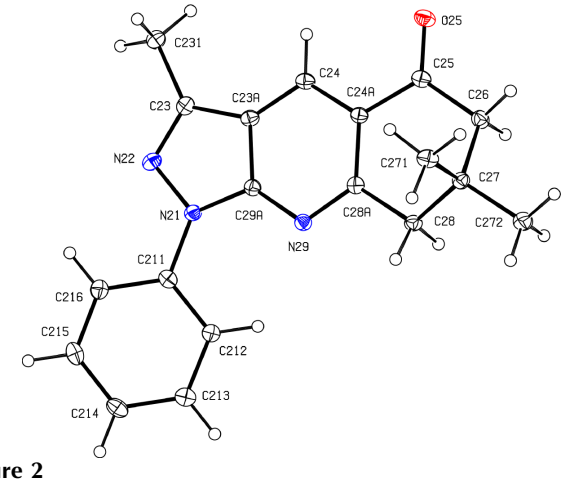


Figure 2
A view of molecule 2 of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

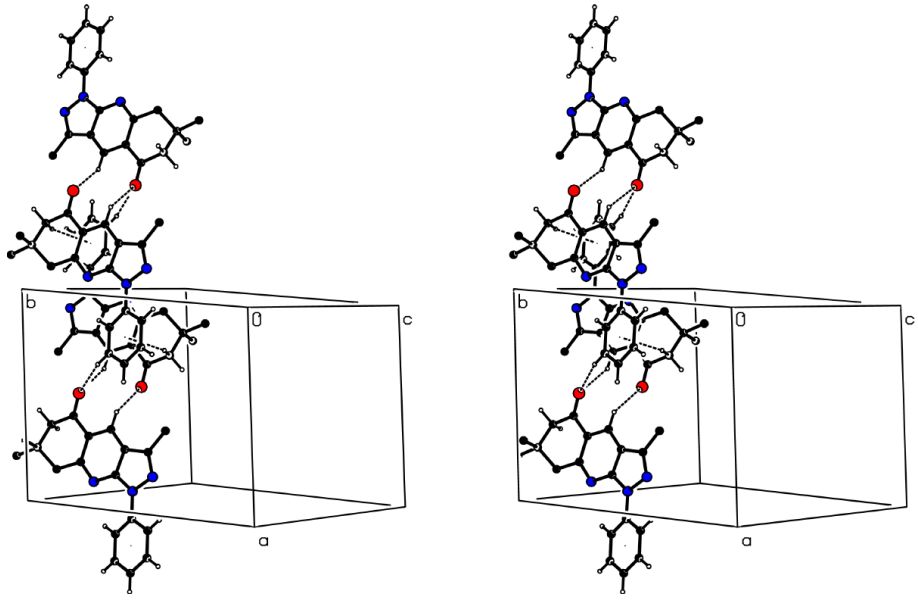


Figure 3
Stereoview of the molecular chains running along the *a* axis, formed by the dimers of molecule 1 linked by C—H···π bonds.

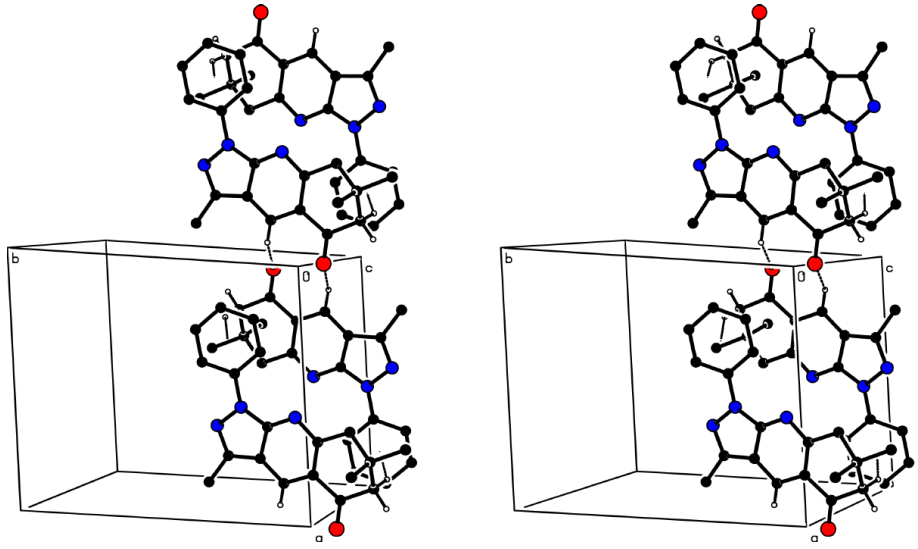


Figure 4
Stereoview of the molecular chains running along the *a* axis, formed by the dimers of molecule 2 linked by C—H···π bonds.

Crystal data

$C_{19}H_{19}N_3O$	$Z = 4$
$M_r = 305.37$	$D_x = 1.312$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo Kα radiation
$a = 10.6825$ (4) Å	Cell parameters from 7071 reflections
$b = 11.9254$ (4) Å	$\theta = 3.2$–$27.6°$
$c = 13.1219$ (6) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 111.2105$ (17)°	$T = 120.0$ (2) K
$\beta = 90.5164$ (18)°	Plate, colourless
$\gamma = 96.533$ (2)°	0.25 × 0.24 × 0.03 mm
$V = 1546.00$ (11) Å^3	

Data collection

Nonius KappaCCD diffractometer	7071 independent reflections
φ scans and ω scans with κ offsets	4952 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)	$R_{int} = 0.080$
	$\theta_{max} = 27.6°$
$T_{min} = 0.980$, $T_{max} = 0.998$	$h = -13 \rightarrow 13$
32382 measured reflections	$k = -15 \rightarrow 15$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.5429P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.059$	
$wR(F^2) = 0.138$	
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
7071 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
421 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected torsion angles (°).

C19*A*—N11—C111—C112	−17.8 (3)	C29*A*—N21—C211—C212	−22.8 (3)
N12—N11—C111—C112	167.34 (16)	N22—N21—C211—C212	163.71 (16)
C19*A*—N11—C111—C116	162.52 (18)	C29*A*—N21—C211—C216	157.20 (18)
N12—N11—C111—C116	−12.3 (2)	N22—N21—C211—C216	−16.2 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C112—H112···N19	0.95	2.38	3.004 (3)	123
C212—H212···N29	0.95	2.40	3.000 (3)	121
C14—H14···O15^{i}	0.95	2.59	3.258 (2)	127
C24—H24···O25^{ii}	0.95	2.54	3.221 (2)	129
C113—H113···O15^{iii}	0.95	2.54	3.461 (3)	164
C16—H16B···*Cg*1^{iv}	0.99	2.77	3.656 (2)	150
C26—H26B···*Cg*2^{v}	0.99	2.90	3.725 (2)	142

Symmetry codes: (i) $1-x, 2-y, 1-z$; (ii) $-x, -y, -z$; (iii) $x-1, y, z$; (iv) $-x, 2-y, 1-z$; (v) $1-x, -y, -z$.

H atoms were treated as riding atoms, with C—H = 0.95 Å (aromatic), 0.98 Å (methyl) and 0.99 Å (CH_2). The starting positions for the methyl H atoms were obtained from a difference map.

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, 2003); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

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References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Cremer, D & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Nonius (1997). *KappaCCD Server Software*. Windows 3.11 Version. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.