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ISSN 2414-3146

3-Methyl-5-(4-methylpiperazin-1-yl)-1-phenyl-1*H*-pyrazole-4-carbaldehyde

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Received 6 October 2016
Accepted 9 October 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

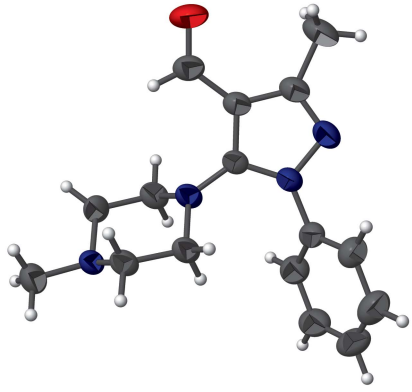
Keywords: crystal structure; pyrazole; hydrogen bonding.

CCDC reference: 1509001

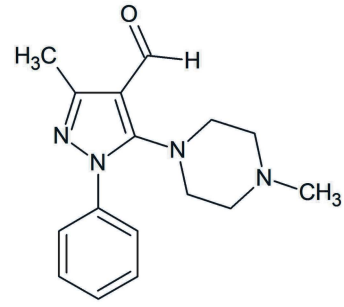
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_{20}N_4O$, the dihedral angle between the pyrazole and phenyl rings is 53.86 (12)°. The piperazine ring adopts a chair conformation with the exocyclic N—C bonds in equatorial orientations. In the crystal, molecules are linked by very weak C—H···O hydrogen bonds to generate [010] $C(8)$ chains, with adjacent molecules related by translation.

3D view



Chemical scheme



Structure description

Piperazine derivatives are found in biologically active compounds across many therapeutic areas and display antipsychotic (Chaudhary *et al.*, 2006) and antifungal (Upadhayaya *et al.*, 2004) behaviours. As part of our ongoing studies in this area (Girisha *et al.*, 2010), herein we report the synthesis and structure of the title compound (Fig. 1).

The dihedral angle between the pyrazole (r.m.s. deviation = 0.012 Å) and phenyl rings is 53.86 (12)°. The piperazine ring adopts a chair conformation with the exocyclic N—C bonds in equatorial orientations. The piperazine ring bisects the plane of the pyrazole ring which is evident from the dihedral angle value of 53.57 (10)° between the mean planes of the pyrazole and piperazine (all atoms) rings.

In the crystal, the molecules are linked by weak C—H···O hydrogen bonds (Table 1) to generate [010] $C(8)$ chains, with adjacent molecules related by translation (Fig. 2).

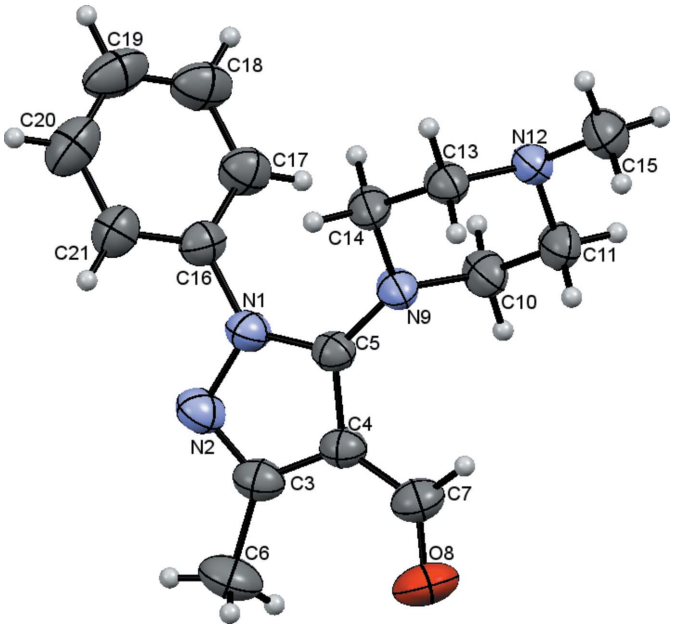


Figure 1
A view of the title compound, with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

Synthesis and crystallization

After synthesis and purification (Girisha *et al.*, 2010), the title compound was dissolved in ethanol and the solution was gently heated and left undisturbed. After slow evaporation of the solvent, colourless blocks grew after 10 d.

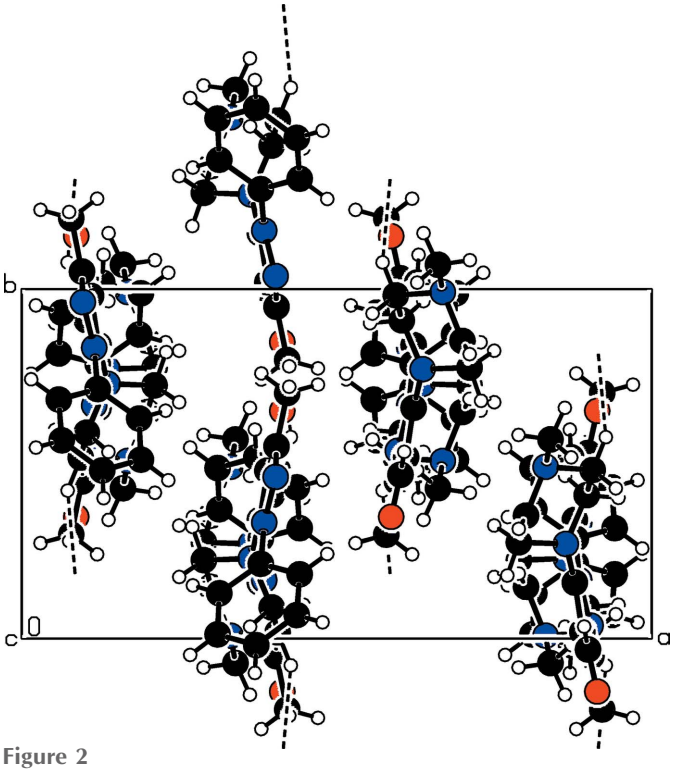


Figure 2
Packing diagram of the title molecule, viewed down the *c* axis. The dashed lines represent hydrogen bonds.

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···O8^{i}	0.97	2.60	3.4753 (19)	151

Symmetry code: (i) $x, y+1, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{20}N_4O$
M_r	284.36
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	293
a, b, c (Å)	17.1027 (13), 9.4885 (8), 18.6837 (16)
V (Å^3)	3032.0 (4)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.65
Crystal size (mm)	0.30 × 0.27 × 0.25
Data collection	
Diffractometer	Bruker X8 Proteum CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.824, 0.851
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14169, 2540, 2027
R_{int}	0.078
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.585
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.117, 1.07
No. of reflections	2540
No. of parameters	193
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.17

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank the Institution of Excellence, Vijnana Bhavana, University of Mysore, Mysuru for providing the single-crystal X-ray diffractometer facility.

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full crystallographic data

IUCrData (2016). **1**, x161593 [https://doi.org/10.1107/S2414314616015935]

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Crystal data

$C_{16}H_{20}N_4O$
M_r = 284.36
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 17.1027 (13) Å
b = 9.4885 (8) Å
c = 18.6837 (16) Å
V = 3032.0 (4) Å^3
Z = 8
F(000) = 1216
D_x = 1.246 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54178 Å
Cell parameters from 2501 reflections
θ = 5.4–64.5°
μ = 0.65 mm^{-1}
T = 293 K
Block, colorless
0.30 × 0.27 × 0.25 mm

Data collection

Bruker X8 Proteum CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.7 pixels mm^{-1}
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.824, T_{max} = 0.851
14169 measured reflections
2540 independent reflections
2027 reflections with $I > 2\sigma(I)$
R_{int} = 0.078
θ_{max} = 64.5°, θ_{min} = 5.4°
h = −20→20
k = −11→10
l = −15→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.117
S = 1.07
2540 reflections
193 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.1549P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0020 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.38513 (6)	0.16761 (12)	0.60098 (8)	0.0434 (3)
N2	0.40285 (7)	0.03759 (14)	0.63048 (8)	0.0514 (4)
C3	0.40416 (7)	−0.05029 (17)	0.57596 (10)	0.0474 (4)
C4	0.38990 (7)	0.01950 (15)	0.51072 (9)	0.0418 (4)
C5	0.37799 (6)	0.16018 (14)	0.52887 (9)	0.0385 (4)
C6	0.42103 (10)	−0.20264 (18)	0.58809 (12)	0.0706 (6)
H6A	0.4270	−0.2198	0.6384	0.106*
H6B	0.4684	−0.2279	0.5637	0.106*
H6C	0.3786	−0.2583	0.5699	0.106*
C7	0.39904 (8)	−0.03138 (17)	0.43884 (10)	0.0524 (5)
H7	0.3933	0.0342	0.4022	0.063*
O8	0.41359 (7)	−0.15167 (12)	0.42117 (8)	0.0698 (4)
N9	0.36366 (6)	0.27099 (12)	0.48325 (7)	0.0427 (3)
C10	0.28918 (7)	0.27548 (16)	0.44428 (9)	0.0485 (4)
H10A	0.2501	0.3235	0.4729	0.058*
H10B	0.2710	0.1803	0.4352	0.058*
C11	0.30008 (8)	0.35158 (16)	0.37487 (10)	0.0506 (4)
H11A	0.3354	0.2984	0.3446	0.061*
H11B	0.2502	0.3588	0.3504	0.061*
N12	0.33155 (6)	0.49159 (12)	0.38653 (7)	0.0417 (3)
C13	0.40635 (7)	0.48233 (16)	0.42350 (9)	0.0449 (4)
H13A	0.4272	0.5763	0.4308	0.054*
H13B	0.4432	0.4300	0.3943	0.054*
C14	0.39691 (8)	0.41040 (16)	0.49448 (9)	0.0481 (4)
H14A	0.4473	0.4022	0.5179	0.058*
H14B	0.3627	0.4655	0.5250	0.058*
C15	0.33932 (9)	0.56944 (18)	0.31972 (10)	0.0586 (5)
H15A	0.3725	0.5181	0.2875	0.088*
H15B	0.3619	0.6602	0.3292	0.088*
H15C	0.2887	0.5813	0.2984	0.088*
C16	0.37949 (7)	0.28622 (16)	0.64745 (9)	0.0441 (4)
C17	0.31364 (8)	0.37036 (18)	0.64642 (10)	0.0541 (5)
H17	0.2717	0.3478	0.6169	0.065*
C18	0.31068 (10)	0.48774 (19)	0.68942 (11)	0.0641 (5)
H18	0.2670	0.5461	0.6882	0.077*

C19	0.37206 (11)	0.51924 (19)	0.73435 (11)	0.0712 (6)
H19	0.3700	0.5991	0.7631	0.085*
C20	0.43651 (10)	0.4323 (2)	0.73658 (10)	0.0679 (5)
H20	0.4773	0.4526	0.7678	0.081*
C21	0.44102 (8)	0.31600 (18)	0.69310 (9)	0.0542 (4)
H21	0.4848	0.2580	0.6943	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0510 (6)	0.0348 (7)	0.0443 (9)	0.0023 (5)	0.0005 (5)	0.0027 (7)
N2	0.0608 (7)	0.0412 (7)	0.0522 (10)	0.0021 (5)	0.0014 (6)	0.0104 (7)
C3	0.0460 (7)	0.0369 (8)	0.0592 (12)	−0.0010 (6)	0.0036 (7)	0.0031 (9)
C4	0.0430 (6)	0.0336 (8)	0.0487 (11)	−0.0025 (5)	0.0012 (6)	−0.0002 (8)
C5	0.0374 (6)	0.0333 (7)	0.0449 (10)	−0.0029 (5)	−0.0012 (6)	0.0007 (8)
C6	0.0827 (10)	0.0402 (9)	0.0888 (17)	0.0069 (8)	0.0030 (10)	0.0140 (11)
C7	0.0571 (7)	0.0400 (9)	0.0599 (13)	−0.0068 (6)	0.0025 (7)	−0.0051 (9)
O8	0.0849 (7)	0.0421 (7)	0.0823 (11)	−0.0028 (5)	0.0116 (7)	−0.0181 (7)
N9	0.0454 (5)	0.0322 (6)	0.0503 (9)	−0.0062 (5)	−0.0128 (5)	0.0032 (6)
C10	0.0425 (6)	0.0428 (8)	0.0602 (11)	−0.0067 (6)	−0.0113 (7)	0.0043 (9)
C11	0.0513 (7)	0.0465 (9)	0.0539 (12)	−0.0036 (6)	−0.0160 (7)	0.0004 (9)
N12	0.0448 (5)	0.0360 (6)	0.0444 (8)	0.0025 (4)	−0.0066 (5)	0.0026 (7)
C13	0.0442 (6)	0.0367 (8)	0.0537 (11)	−0.0041 (6)	−0.0084 (6)	0.0036 (8)
C14	0.0564 (7)	0.0353 (8)	0.0526 (11)	−0.0091 (6)	−0.0155 (7)	0.0030 (9)
C15	0.0707 (9)	0.0535 (9)	0.0516 (11)	0.0013 (7)	−0.0105 (8)	0.0085 (10)
C16	0.0518 (7)	0.0412 (8)	0.0393 (9)	−0.0028 (6)	0.0083 (6)	0.0001 (8)
C17	0.0532 (7)	0.0559 (10)	0.0531 (11)	0.0049 (7)	0.0080 (7)	−0.0026 (10)
C18	0.0740 (9)	0.0576 (10)	0.0606 (13)	0.0113 (8)	0.0231 (9)	−0.0026 (11)
C19	0.0965 (13)	0.0579 (10)	0.0590 (14)	−0.0083 (9)	0.0274 (11)	−0.0154 (11)
C20	0.0754 (10)	0.0754 (12)	0.0529 (12)	−0.0176 (9)	0.0050 (9)	−0.0138 (12)
C21	0.0562 (7)	0.0587 (9)	0.0477 (11)	−0.0031 (7)	0.0002 (7)	−0.0040 (10)

Geometric parameters (Å, º)

N1—C5	1.354 (2)	N12—C15	1.457 (2)
N1—N2	1.3848 (16)	N12—C13	1.4566 (16)
N1—C16	1.4247 (19)	C13—C14	1.500 (2)
N2—C3	1.317 (2)	C13—H13A	0.9700
C3—C4	1.408 (2)	C13—H13B	0.9700
C3—C6	1.491 (2)	C14—H14A	0.9700
C4—C5	1.3921 (18)	C14—H14B	0.9700
C4—C7	1.436 (2)	C15—H15A	0.9600
C5—N9	1.3756 (18)	C15—H15B	0.9600
C6—H6A	0.9600	C15—H15C	0.9600
C6—H6B	0.9600	C16—C17	1.381 (2)
C6—H6C	0.9600	C16—C21	1.384 (2)
C7—O8	1.2140 (18)	C17—C18	1.374 (2)
C7—H7	0.9300	C17—H17	0.9300

N9—C14	1.4551 (17)	C18—C19	1.377 (3)
N9—C10	1.4678 (16)	C18—H18	0.9300
C10—C11	1.496 (2)	C19—C20	1.377 (3)
C10—H10A	0.9700	C19—H19	0.9300
C10—H10B	0.9700	C20—C21	1.373 (2)
C11—N12	1.4498 (18)	C20—H20	0.9300
C11—H11A	0.9700	C21—H21	0.9300
C11—H11B	0.9700		

C5—N1—N2	111.67 (12)	C11—N12—C13	110.00 (11)
C5—N1—C16	129.89 (13)	C15—N12—C13	110.91 (11)
N2—N1—C16	118.42 (14)	N12—C13—C14	110.61 (11)
C3—N2—N1	105.07 (14)	N12—C13—H13A	109.5
N2—C3—C4	111.64 (14)	C14—C13—H13A	109.5
N2—C3—C6	119.98 (17)	N12—C13—H13B	109.5
C4—C3—C6	128.37 (17)	C14—C13—H13B	109.5
C5—C4—C3	105.38 (15)	H13A—C13—H13B	108.1
C5—C4—C7	124.49 (15)	N9—C14—C13	109.16 (13)
C3—C4—C7	129.24 (14)	N9—C14—H14A	109.8
N1—C5—N9	126.34 (13)	C13—C14—H14A	109.8
N1—C5—C4	106.21 (13)	N9—C14—H14B	109.8
N9—C5—C4	127.44 (16)	C13—C14—H14B	109.8
C3—C6—H6A	109.5	H14A—C14—H14B	108.3
C3—C6—H6B	109.5	N12—C15—H15A	109.5
H6A—C6—H6B	109.5	N12—C15—H15B	109.5
C3—C6—H6C	109.5	H15A—C15—H15B	109.5
H6A—C6—H6C	109.5	N12—C15—H15C	109.5
H6B—C6—H6C	109.5	H15A—C15—H15C	109.5
O8—C7—C4	126.37 (17)	H15B—C15—H15C	109.5
O8—C7—H7	116.8	C17—C16—C21	120.72 (16)
C4—C7—H7	116.8	C17—C16—N1	120.23 (14)
C5—N9—C14	122.40 (12)	C21—C16—N1	119.05 (13)
C5—N9—C10	118.97 (10)	C18—C17—C16	119.38 (16)
C14—N9—C10	112.60 (11)	C18—C17—H17	120.3
N9—C10—C11	109.62 (11)	C16—C17—H17	120.3
N9—C10—H10A	109.7	C17—C18—C19	120.28 (16)
C11—C10—H10A	109.7	C17—C18—H18	119.9
N9—C10—H10B	109.7	C19—C18—H18	119.9
C11—C10—H10B	109.7	C18—C19—C20	119.90 (18)
H10A—C10—H10B	108.2	C18—C19—H19	120.0
N12—C11—C10	111.00 (13)	C20—C19—H19	120.0
N12—C11—H11A	109.4	C21—C20—C19	120.57 (17)
C10—C11—H11A	109.4	C21—C20—H20	119.7
N12—C11—H11B	109.4	C19—C20—H20	119.7
C10—C11—H11B	109.4	C20—C21—C16	119.10 (15)
H11A—C11—H11B	108.0	C20—C21—H21	120.4
C11—N12—C15	111.71 (13)	C16—C21—H21	120.4

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···O8^{i}	0.97	2.60	3.4753 (19)	151

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