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(*E*)-2-[(4-Chloro-1,3-dimethyl-1*H*-pyrazol-5-yl)methyleneamino]benzamide

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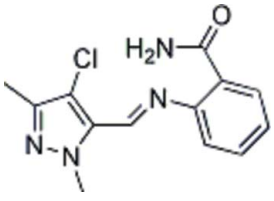
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.098; data-to-parameter ratio = 13.6.

In the title compound, $C_{13}H_{13}ClN_4O$, the dihedral angle between the aromatic rings is 33.47 (9)° and an intramolecular N—H···N hydrogen bond generates an S(6) ring. In the crystal, inversion dimers linked by pairs of N—H···O hydrogen bonds occur, resulting in $R_2^2(8)$ loops.

Related literature

For catalytic studies on a related compound, see: Chen *et al.* (2008).



Experimental

Crystal data

$C_{13}H_{13}ClN_4O$ M_r = 276.72
Monoclinic, $C2/c$
a = 22.046 (3) Å
b = 8.6785 (10) Å
c = 14.1137 (16) Å
β = 96.005 (2)°
V = 2685.5 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 296 K
0.32 × 0.30 × 0.28 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.725, T_{max} = 1.000
6634 measured reflections
2375 independent reflections
2098 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.098
S = 1.06
2375 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4B···N3	0.86	2.07	2.749 (2)	136
N4—H4A···O1^{i}	0.86	2.10	2.930 (2)	161

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5252).

References

Bruker (2001). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, J., Su, W., Wu, H., Liu, M. & Jin, C. (2008). *Catal. Commun.* **9**, 785–788.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2009). E**65**, o3263 [**doi:10.1107/S1600536809050685**]

(*E*)-2-[(4-Chloro-1,3-dimethyl-1*H*-pyrazol-5-yl)methyleneamino]benzamide

Y. Cong, H. Yang, H. Yu and B. Li

Experimental

The title compound was synthesized by the reaction of 4-chloro-1,3-dimethyl-1*H*-pyrazole-5- carbaldehyde with 2-aminobenzamide in 1,2-dichloroethane. The crude product was purified by silica-gel column chromatography and then grown from methylene chloride to afford colourless blocks of (I).

Anal. Calcd for $C_{13}H_{13}N_4O_1$: C, 56.42; H, 4.74; N, 20.25; O, 5.78. Found: C, 56.57; H, 4.64; N, 20.66; O, 5.25. ^{1}H NMR(DMSO): 2.21(s,3*H*, CH_3), 4.12 (s,3*H*, N—CH_3), 7.33(s, 2H, $CONH_2$), 7.50–7.61 (m, 2H, Ph), 7.89 (t, 1H, Ph—H), 8.26 (s, 1H, Ph—H), 8.46 (s, 1H, CH=N).

Refinement

All H atoms were visible in difference maps; they were finally placed in geometrically calculated positions, with C—H = 0.93–0.96Å and N—H = 0.86 Å, and refined as riding with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(methyl C).

Figures

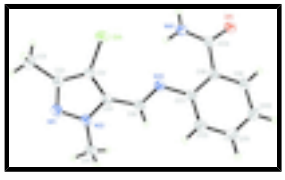


Fig. 1. The molecular structure of (I), with 30% probability displacement ellipsoids.

(*E*)-2-[(4-Chloro-1,3-dimethyl-1*H*-pyrazol-5- yl)methyleneamino]benzamide

Crystal data

$C_{13}H_{13}ClN_4O$	F(000) = 1152
M_r = 276.72	D_x = 1.369 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 4144 reflections
a = 22.046 (3) Å	θ = 2.2–30.2°
b = 8.6785 (10) Å	μ = 0.28 mm^{-1}
c = 14.1137 (16) Å	T = 296 K
β = 96.005 (2)°	Block, colourless
V = 2685.5 (5) Å^3	0.32 × 0.30 × 0.28 mm
Z = 8	

Data collection

Bruker SMART CCD diffractometer	2375 independent reflections
Radiation source: fine-focus sealed tube	2098 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.018$
ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 1.9°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	$h = -25\rightarrow26$
$T_{min} = 0.725$, $T_{max} = 1.000$	$k = -6\rightarrow10$
6634 measured reflections	$l = -16\rightarrow16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 1.4091P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2375 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
175 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0266 (12)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.45129 (2)	0.49955 (4)	0.12551 (4)	0.0612 (2)
N4	0.29251 (6)	0.43146 (17)	0.04310 (11)	0.0531 (4)
H4A	0.3006	0.3480	0.0140	0.064*
H4B	0.3217	0.4899	0.0670	0.064*
O1	0.19254 (5)	0.38809 (14)	0.01737 (10)	0.0600 (4)
N1	0.52045 (6)	0.91074 (16)	0.12334 (10)	0.0486 (4)
N2	0.46079 (6)	0.94242 (16)	0.12143 (10)	0.0445 (3)
C6	0.36052 (7)	0.82150 (17)	0.12489 (11)	0.0397 (4)
H6	0.3417	0.9174	0.1202	0.048*

N3	0.32795 (6)	0.70280 (14)	0.13302 (9)	0.0376 (3)
C1	0.46698 (7)	0.69287 (17)	0.12368 (11)	0.0400 (4)
C2	0.52480 (7)	0.75722 (19)	0.12428 (11)	0.0430 (4)
C3	0.58468 (8)	0.6779 (2)	0.12766 (15)	0.0576 (5)
H3A	0.6159	0.7518	0.1181	0.086*
H3B	0.5831	0.6013	0.0784	0.086*
H3C	0.5938	0.6294	0.1886	0.086*
C4	0.44200 (9)	1.1028 (2)	0.12241 (16)	0.0614 (5)
H4C	0.4316	1.1292	0.1847	0.092*
H4D	0.4072	1.1179	0.0766	0.092*
H4E	0.4749	1.1672	0.1066	0.092*
C5	0.42578 (7)	0.81350 (17)	0.12263 (11)	0.0379 (4)
C7	0.26516 (7)	0.72357 (17)	0.14214 (10)	0.0364 (3)
C8	0.22185 (7)	0.61479 (17)	0.10333 (10)	0.0363 (3)
C9	0.16068 (7)	0.6395 (2)	0.11507 (12)	0.0445 (4)
H9	0.1317	0.5694	0.0890	0.053*
C10	0.14195 (8)	0.7653 (2)	0.16439 (13)	0.0541 (5)
H10	0.1007	0.7807	0.1701	0.065*
C11	0.18475 (8)	0.8675 (2)	0.20506 (14)	0.0577 (5)
H11	0.1726	0.9509	0.2398	0.069*
C12	0.24531 (8)	0.8468 (2)	0.19446 (13)	0.0504 (4)
H12	0.2738	0.9163	0.2227	0.060*
C13	0.23540 (7)	0.46978 (18)	0.05100 (11)	0.0401 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0557 (3)	0.0314 (3)	0.0956 (4)	−0.00049 (17)	0.0037 (2)	−0.0028 (2)
N4	0.0376 (7)	0.0445 (8)	0.0774 (10)	−0.0038 (6)	0.0064 (7)	−0.0244 (7)
O1	0.0391 (6)	0.0426 (7)	0.0960 (10)	−0.0043 (5)	−0.0039 (6)	−0.0204 (6)
N1	0.0387 (7)	0.0446 (8)	0.0618 (9)	−0.0076 (6)	0.0021 (6)	0.0017 (6)
N2	0.0407 (7)	0.0328 (7)	0.0589 (8)	−0.0046 (5)	−0.0004 (6)	0.0008 (6)
C6	0.0404 (8)	0.0316 (8)	0.0452 (8)	0.0002 (6)	−0.0044 (6)	0.0003 (6)
N3	0.0356 (7)	0.0345 (7)	0.0418 (7)	−0.0026 (5)	−0.0006 (5)	−0.0032 (5)
C1	0.0417 (8)	0.0332 (8)	0.0442 (8)	−0.0017 (6)	−0.0001 (6)	−0.0015 (6)
C2	0.0403 (8)	0.0434 (9)	0.0450 (9)	−0.0012 (7)	0.0025 (7)	−0.0004 (7)
C3	0.0447 (10)	0.0609 (12)	0.0674 (12)	0.0047 (8)	0.0066 (8)	0.0000 (9)
C4	0.0561 (11)	0.0322 (9)	0.0946 (15)	−0.0052 (8)	0.0017 (10)	−0.0010 (9)
C5	0.0389 (8)	0.0322 (8)	0.0414 (8)	−0.0043 (6)	−0.0018 (6)	−0.0007 (6)
C7	0.0374 (8)	0.0346 (8)	0.0364 (7)	0.0027 (6)	0.0004 (6)	0.0009 (6)
C8	0.0367 (8)	0.0338 (8)	0.0381 (8)	−0.0006 (6)	0.0018 (6)	0.0023 (6)
C9	0.0366 (8)	0.0452 (9)	0.0513 (9)	−0.0019 (7)	0.0024 (7)	0.0001 (7)
C10	0.0404 (9)	0.0613 (11)	0.0614 (11)	0.0102 (8)	0.0097 (8)	−0.0044 (9)
C11	0.0547 (11)	0.0574 (11)	0.0608 (11)	0.0142 (9)	0.0046 (8)	−0.0175 (9)
C12	0.0478 (10)	0.0461 (9)	0.0557 (10)	0.0014 (8)	−0.0012 (8)	−0.0145 (8)
C13	0.0379 (8)	0.0343 (8)	0.0472 (8)	−0.0027 (6)	0.0009 (6)	−0.0010 (6)

Geometric parameters (Å, °)

Cl1—C1	1.7137 (16)	C3—H3B	0.9600
N4—C13	1.318 (2)	C3—H3C	0.9600
N4—H4A	0.8600	C4—H4C	0.9600
N4—H4B	0.8600	C4—H4D	0.9600
O1—C13	1.2356 (18)	C4—H4E	0.9600
N1—C2	1.336 (2)	C7—C12	1.396 (2)
N1—N2	1.3413 (19)	C7—C8	1.412 (2)
N2—C5	1.360 (2)	C8—C9	1.392 (2)
N2—C4	1.452 (2)	C8—C13	1.505 (2)
C6—N3	1.2677 (19)	C9—C10	1.381 (2)
C6—C5	1.444 (2)	C9—H9	0.9300
C6—H6	0.9300	C10—C11	1.376 (3)
N3—C7	1.4151 (19)	C10—H10	0.9300
C1—C5	1.385 (2)	C11—C12	1.371 (2)
C1—C2	1.391 (2)	C11—H11	0.9300
C2—C3	1.485 (2)	C12—H12	0.9300
C3—H3A	0.9600		
C13—N4—H4A	120.0	H4C—C4—H4E	109.5
C13—N4—H4B	120.0	H4D—C4—H4E	109.5
H4A—N4—H4B	120.0	N2—C5—C1	104.45 (14)
C2—N1—N2	105.89 (13)	N2—C5—C6	121.91 (13)
N1—N2—C5	112.81 (13)	C1—C5—C6	133.62 (14)
N1—N2—C4	118.44 (13)	C12—C7—C8	118.74 (14)
C5—N2—C4	128.70 (14)	C12—C7—N3	120.63 (13)
N3—C6—C5	122.55 (14)	C8—C7—N3	120.52 (13)
N3—C6—H6	118.7	C9—C8—C7	118.35 (14)
C5—C6—H6	118.7	C9—C8—C13	115.57 (13)
C6—N3—C7	118.26 (13)	C7—C8—C13	126.07 (13)
C5—C1—C2	107.22 (14)	C10—C9—C8	121.76 (16)
C5—C1—Cl1	127.38 (12)	C10—C9—H9	119.1
C2—C1—Cl1	125.40 (13)	C8—C9—H9	119.1
N1—C2—C1	109.62 (14)	C11—C10—C9	119.45 (16)
N1—C2—C3	121.67 (15)	C11—C10—H10	120.3
C1—C2—C3	128.70 (16)	C9—C10—H10	120.3
C2—C3—H3A	109.5	C12—C11—C10	120.20 (16)
C2—C3—H3B	109.5	C12—C11—H11	119.9
H3A—C3—H3B	109.5	C10—C11—H11	119.9
C2—C3—H3C	109.5	C11—C12—C7	121.41 (16)
H3A—C3—H3C	109.5	C11—C12—H12	119.3
H3B—C3—H3C	109.5	C7—C12—H12	119.3
N2—C4—H4C	109.5	O1—C13—N4	121.44 (15)
N2—C4—H4D	109.5	O1—C13—C8	119.04 (14)
H4C—C4—H4D	109.5	N4—C13—C8	119.52 (13)
N2—C4—H4E	109.5		
C2—N1—N2—C5	0.99 (18)	N3—C6—C5—C1	3.3 (3)
C2—N1—N2—C4	178.50 (16)	C6—N3—C7—C12	−37.2 (2)

C5—C6—N3—C7	175.07 (13)	C6—N3—C7—C8	146.70 (15)
N2—N1—C2—C1	−0.46 (18)	C12—C7—C8—C9	3.1 (2)
N2—N1—C2—C3	−179.23 (15)	N3—C7—C8—C9	179.31 (13)
C5—C1—C2—N1	−0.19 (18)	C12—C7—C8—C13	−175.57 (15)
Cl1—C1—C2—N1	−179.43 (12)	N3—C7—C8—C13	0.6 (2)
C5—C1—C2—C3	178.46 (17)	C7—C8—C9—C10	−0.9 (2)
Cl1—C1—C2—C3	−0.8 (3)	C13—C8—C9—C10	177.86 (15)
N1—N2—C5—C1	−1.09 (18)	C8—C9—C10—C11	−1.5 (3)
C4—N2—C5—C1	−178.29 (17)	C9—C10—C11—C12	1.7 (3)
N1—N2—C5—C6	177.42 (13)	C10—C11—C12—C7	0.5 (3)
C4—N2—C5—C6	0.2 (3)	C8—C7—C12—C11	−2.9 (3)
C2—C1—C5—N2	0.75 (17)	N3—C7—C12—C11	−179.15 (16)
Cl1—C1—C5—N2	179.96 (12)	C9—C8—C13—O1	4.2 (2)
C2—C1—C5—C6	−177.51 (16)	C7—C8—C13—O1	−177.06 (15)
Cl1—C1—C5—C6	1.7 (3)	C9—C8—C13—N4	−174.85 (15)
N3—C6—C5—N2	−174.72 (15)	C7—C8—C13—N4	3.9 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4B···N3	0.86	2.07	2.749 (2)	136
N4—H4A···O1^{i}	0.86	2.10	2.930 (2)	161

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

Fig. 1