Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-*N*-[(5-Chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]aniline

Feng-Mei Sun,[a]* Li Yang,[b] Xiao-Bao Chen[c] and Zhao-Xun Lian[a]

[a]School of Chemistry and Chemical Engineering, Henan Institute of Science and Technology, Xinxiang 453003, Henan, People's Republic of China, [b]Experimental Center, Henan Institute of Science and Technology, Xinxiang 453003, Henan, People's Republic of China, and [c]Department of Medicinal Chemistry, Yunyang Medical College, Shiyan 442000, Hubei, People's Republic of China
Correspondence e-mail: sfmmm1219@126.com

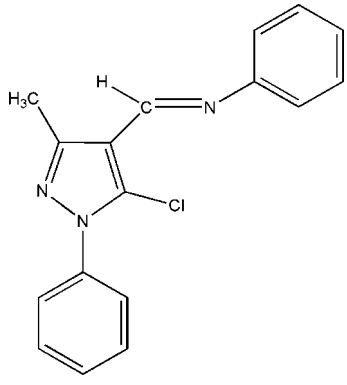
Received 9 September 2007; accepted 1 November 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.091; data-to-parameter ratio = 14.4.

The title compound, $C_{17}H_{14}ClN_3$, adopts the more stable E configuration. In the crystal structure, the pyrazole heterocycle and the adjacent benzene ring are not coplanar but inclined at 45.54 (7)° . The imine group is twisted by 5.41 (12)° away from the pyrazole ring, and by 42.59 (11)° away from the adjacent benzene ring.

Related literature

Many derivatives of pyrazole have been prepared, and their biological activities have been studied by Wang (2004), He (2005) and Liang & Bai (2006). A closely related structure was published by Trilleras *et al.* (2005).



Experimental

Crystal data

$C_{17}H_{14}ClN_3$
M_r = 295.76
Monoclinic, $P2_1/n$
a = 8.7837 (9) Å
b = 11.0980 (12) Å
c = 15.6749 (16) Å
β = 102.6480 (10)°
V = 1490.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 (2) K
0.40 × 0.19 × 0.18 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.816, T_{max} = 0.955
9958 measured reflections
2751 independent reflections
2248 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.05
2751 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *APEX2* (Bruker 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker 2004); software used to prepare material for publication: *SHELXTL*.

The authors thank the Henan Institute of Science and Technology for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2071).

References

Bruker (2004). *APEXII* (Version 1.027), *SAINT* (Version 7.12A) and *SHELXTL* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
He, H.-W. (2005). *Pesticides*, **44**, 491–495.
Liang, Y. & Bai, Y.-L. (2006). *Mod. Agrochem.* **5**, 6–12.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Trilleras, J., Quiroga, J., Cobo, J., Low, J. N. & Glidewell, C. (2005). *Acta Cryst.* E**61**, o1055–o1057.
Wang, H.-Q. (2004). PhD thesis, Central China Normal University, People's Republic of China, pp. 1–18.

  doi:10.1107/S1600536807055390

supplementary materials

Acta Cryst. (2007). E**63**, o4620 [**doi:10.1107/S1600536807055390**]

(*E*)-*N*-[(5-Chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]aniline

F.-M. Sun, L. Yang, X.-B. Chen and Z.-X. Lian

Comment

Pyrazole derivatives are attracting the increasing attention of the synthetic community for decades (Wang, 2004), owing to their wide range of biological activities in pesticide and medicine science (Liang & Bai, 2006; He, 2005). In recent years, we have been engaged in the preparation of derivatives of pyrazole compounds, and expected to find low toxicity and high activity lead compounds for the pesticide field.

Herein, we report the crystal structure of the title compound (Fig. 1), which was synthesized by the condensation reaction of aldehyde with aniline.

The title compound adopts the more stable E-configuration. In the pyrazole ring of the title molecule, bond lengths and angles are similar to those observed in closely related structures (Trilleras *et al.*, 2005). The pyrazole ring and the adjacent benzene ring (C1–C6) are inclined at 45.54 (7)°. The imine group (N3—C11—H11) is twisted by 5.41 (12)° away from the pyrazole ring, and 42.59 (11)° away from the adjacent benzene ring (C12–C17).

Experimental

A solution of 5-chloro-3-methyl-1-*H*-pyrazole-4-carbalhedyde (5 mmol) and aniline (6 mmol) in anhydrous ethanol (20 ml) were stirred under reflux until the reaction was completed (monitored by thin-layer chromatography). After removal of ethanol under reduced pressure, the residue was recrystallized from ethanol to give the target compound as a yellow solid (yield: 86%, m.p. 395–397 K). A crystal grown from anhydrous ethanol was selected for X-ray structure analysis.

Refinement

H atoms bonded to C were placed at calculated positions, with C—H distances of 0.97 and 0.93Å for H atoms bonded to sp^3 and sp^2 C atoms, respectively. They were refined using a riding model, with U_{iso}(H)=1.2U_{eq}(C),but the methyl H atoms with U_{iso}(H)=1.5U_{eq}(C).

Figures

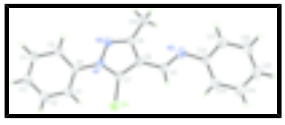


Fig. 1. A view of the molecular structure of the title compound with atom labels and 50% probability displacement ellipsoids for non-H atoms.

(*E*)—*N*-[(5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene]benzenamine

Crystal data

$C_{17}H_{14}ClN_3$	$F_{000} = 616$
$M_r = 295.76$	$D_x = 1.318$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.7837$ (9) Å	Cell parameters from 9958 reflections
$b = 11.0980$ (12) Å	$\theta = 12–18°$
$c = 15.6749$ (16) Å	$\mu = 0.25$ mm^{-1}
$\beta = 102.6480$ (10)°	$T = 293$ (2) K
$V = 1490.9$ (3) Å^3	Block, yellow
$Z = 4$	0.40 × 0.19 × 0.18 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	2248 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.021$
Monochromator: graphite	$\theta_{max} = 25.5°$
φ and ω scans	$\theta_{min} = 2.7°$
Absorption correction: Multi-Scan (SADABS; Sheldrick, 1996)	$h = -10\rightarrow10$
$T_{min} = 0.816$, $T_{max} = 0.955$	$k = -13\rightarrow13$
9958 measured reflections	$l = -18\rightarrow18$
2751 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.034$	H-atom parameters constrained
$wR(F^2) = 0.091$	$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.4599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.002$
2751 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
191 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.63039 (5)	0.64323 (4)	1.01319 (3)	0.05415 (16)
N1	0.74453 (16)	0.43848 (11)	1.09347 (8)	0.0404 (3)
N2	0.82316 (17)	0.33487 (12)	1.08197 (9)	0.0440 (3)
N3	0.86228 (18)	0.43674 (13)	0.82194 (9)	0.0515 (4)
C1	0.7028 (2)	0.55738 (16)	1.21758 (12)	0.0531 (5)
H1	0.7539	0.6225	1.1991	0.064*
C2	0.6436 (3)	0.56587 (18)	1.29239 (12)	0.0633 (5)
H2	0.6532	0.6377	1.3237	0.076*
C3	0.5710 (3)	0.46867 (18)	1.32053 (13)	0.0627 (5)
H3	0.5322	0.4749	1.3710	0.075*
C4	0.5553 (2)	0.36212 (17)	1.27452 (12)	0.0558 (5)
H4	0.5069	0.2964	1.2941	0.067*
C5	0.6118 (2)	0.35303 (15)	1.19894 (11)	0.0466 (4)
H5	0.6007	0.2815	1.1672	0.056*
C6	0.68477 (19)	0.45104 (14)	1.17102 (10)	0.0406 (4)
C7	0.73015 (19)	0.50980 (14)	1.02245 (10)	0.0402 (4)
C8	0.80029 (19)	0.45418 (15)	0.96253 (10)	0.0409 (4)
C9	0.85765 (19)	0.34494 (15)	1.00415 (10)	0.0418 (4)
C10	0.9500 (2)	0.24798 (17)	0.97332 (12)	0.0559 (5)
H10A	0.9787	0.1883	1.0183	0.084*
H10B	1.0425	0.2820	0.9599	0.084*
H10C	0.8883	0.2111	0.9218	0.084*
C11	0.8068 (2)	0.49883 (16)	0.87614 (11)	0.0454 (4)
H11	0.7690	0.5757	0.8600	0.055*
C12	0.8679 (2)	0.48835 (16)	0.73967 (11)	0.0470 (4)
C13	0.9156 (2)	0.60538 (18)	0.73050 (12)	0.0540 (5)
H13	0.9405	0.6556	0.7790	0.065*
C14	0.9263 (3)	0.6478 (2)	0.64888 (13)	0.0655 (6)
H14	0.9594	0.7264	0.6431	0.079*
C15	0.8887 (3)	0.5751 (2)	0.57643 (13)	0.0687 (6)
H15	0.8960	0.6043	0.5219	0.082*
C16	0.8404 (3)	0.4591 (2)	0.58506 (13)	0.0685 (6)
H16	0.8134	0.4100	0.5360	0.082*
C17	0.8315 (2)	0.41479 (19)	0.66612 (12)	0.0604 (5)
H17	0.8010	0.3354	0.6716	0.072*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0622 (3)	0.0463 (3)	0.0581 (3)	0.0086 (2)	0.0222 (2)	0.0086 (2)
N1	0.0501 (8)	0.0379 (7)	0.0369 (7)	−0.0010 (6)	0.0173 (6)	0.0002 (6)
N2	0.0564 (9)	0.0391 (8)	0.0394 (8)	0.0010 (6)	0.0168 (7)	0.0005 (6)
N3	0.0644 (10)	0.0532 (9)	0.0419 (8)	0.0009 (7)	0.0223 (7)	0.0059 (7)
C1	0.0696 (12)	0.0448 (10)	0.0489 (10)	−0.0097 (9)	0.0220 (9)	−0.0056 (8)
C2	0.0918 (16)	0.0539 (12)	0.0504 (11)	−0.0034 (11)	0.0293 (11)	−0.0144 (9)
C3	0.0837 (15)	0.0666 (13)	0.0474 (11)	0.0015 (11)	0.0351 (10)	−0.0037 (9)
C4	0.0721 (13)	0.0549 (11)	0.0477 (10)	−0.0054 (9)	0.0289 (9)	0.0048 (9)
C5	0.0605 (11)	0.0423 (9)	0.0411 (9)	−0.0021 (8)	0.0196 (8)	−0.0001 (7)
C6	0.0477 (10)	0.0435 (9)	0.0330 (8)	0.0000 (7)	0.0140 (7)	0.0006 (7)
C7	0.0437 (9)	0.0381 (9)	0.0406 (9)	−0.0039 (7)	0.0134 (7)	0.0032 (7)
C8	0.0433 (9)	0.0443 (9)	0.0379 (8)	−0.0059 (7)	0.0151 (7)	0.0004 (7)
C9	0.0474 (10)	0.0422 (9)	0.0387 (9)	−0.0036 (7)	0.0156 (7)	−0.0012 (7)
C10	0.0671 (12)	0.0535 (11)	0.0542 (11)	0.0063 (9)	0.0285 (9)	0.0004 (9)
C11	0.0483 (10)	0.0474 (10)	0.0434 (9)	−0.0016 (8)	0.0161 (8)	0.0069 (8)
C12	0.0489 (10)	0.0554 (11)	0.0406 (9)	0.0078 (8)	0.0185 (8)	0.0078 (8)
C13	0.0572 (11)	0.0635 (12)	0.0431 (10)	−0.0049 (9)	0.0149 (8)	0.0047 (9)
C14	0.0731 (14)	0.0708 (14)	0.0569 (12)	−0.0066 (11)	0.0240 (10)	0.0171 (10)
C15	0.0809 (15)	0.0877 (16)	0.0448 (11)	0.0160 (13)	0.0295 (10)	0.0163 (11)
C16	0.0896 (16)	0.0768 (15)	0.0441 (11)	0.0179 (12)	0.0257 (10)	−0.0026 (10)
C17	0.0789 (14)	0.0554 (11)	0.0529 (11)	0.0069 (10)	0.0279 (10)	0.0002 (9)

Geometric parameters (Å, °)

Cl1—C7	1.7106 (17)	C8—C9	1.416 (2)
N1—C7	1.349 (2)	C8—C11	1.455 (2)
N1—N2	1.3734 (18)	C9—C10	1.490 (2)
N1—C6	1.4324 (19)	C10—H10A	0.9600
N2—C9	1.324 (2)	C10—H10B	0.9600
N3—C11	1.271 (2)	C10—H10C	0.9600
N3—C12	1.422 (2)	C11—H11	0.9300
C1—C6	1.378 (2)	C12—C13	1.382 (3)
C1—C2	1.386 (2)	C12—C17	1.392 (3)
C1—H1	0.9300	C13—C14	1.386 (2)
C2—C3	1.374 (3)	C13—H13	0.9300
C2—H2	0.9300	C14—C15	1.373 (3)
C3—C4	1.376 (3)	C14—H14	0.9300
C3—H3	0.9300	C15—C16	1.371 (3)
C4—C5	1.384 (2)	C15—H15	0.9300
C4—H4	0.9300	C16—C17	1.381 (3)
C5—C6	1.382 (2)	C16—H16	0.9300
C5—H5	0.9300	C17—H17	0.9300
C7—C8	1.377 (2)		
C7—N1—N2	109.99 (13)	N2—C9—C10	119.23 (15)

C7—N1—C6	131.12 (14)	C8—C9—C10	129.25 (15)
N2—N1—C6	118.76 (12)	C9—C10—H10A	109.5
C9—N2—N1	105.86 (13)	C9—C10—H10B	109.5
C11—N3—C12	118.84 (15)	H10A—C10—H10B	109.5
C6—C1—C2	119.03 (17)	C9—C10—H10C	109.5
C6—C1—H1	120.5	H10A—C10—H10C	109.5
C2—C1—H1	120.5	H10B—C10—H10C	109.5
C3—C2—C1	120.31 (18)	N3—C11—C8	122.45 (16)
C3—C2—H2	119.8	N3—C11—H11	118.8
C1—C2—H2	119.8	C8—C11—H11	118.8
C2—C3—C4	120.43 (17)	C13—C12—C17	119.01 (16)
C2—C3—H3	119.8	C13—C12—N3	123.00 (16)
C4—C3—H3	119.8	C17—C12—N3	117.90 (17)
C3—C4—C5	119.84 (17)	C12—C13—C14	119.93 (18)
C3—C4—H4	120.1	C12—C13—H13	120.0
C5—C4—H4	120.1	C14—C13—H13	120.0
C6—C5—C4	119.45 (16)	C15—C14—C13	120.7 (2)
C6—C5—H5	120.3	C15—C14—H14	119.6
C4—C5—H5	120.3	C13—C14—H14	119.6
C1—C6—C5	120.92 (15)	C16—C15—C14	119.58 (18)
C1—C6—N1	120.77 (15)	C16—C15—H15	120.2
C5—C6—N1	118.29 (14)	C14—C15—H15	120.2
N1—C7—C8	108.96 (14)	C15—C16—C17	120.4 (2)
N1—C7—Cl1	122.11 (12)	C15—C16—H16	119.8
C8—C7—Cl1	128.87 (13)	C17—C16—H16	119.8
C7—C8—C9	103.69 (14)	C16—C17—C12	120.3 (2)
C7—C8—C11	126.65 (16)	C16—C17—H17	119.9
C9—C8—C11	129.64 (15)	C12—C17—H17	119.9
N2—C9—C8	111.50 (14)		
C7—N1—N2—C9	0.75 (18)	Cl1—C7—C8—C11	0.9 (3)
C6—N1—N2—C9	176.95 (14)	N1—N2—C9—C8	−0.95 (18)
C6—C1—C2—C3	1.3 (3)	N1—N2—C9—C10	177.53 (15)
C1—C2—C3—C4	−0.4 (3)	C7—C8—C9—N2	0.79 (19)
C2—C3—C4—C5	−0.6 (3)	C11—C8—C9—N2	−177.42 (16)
C3—C4—C5—C6	0.6 (3)	C7—C8—C9—C10	−177.49 (18)
C2—C1—C6—C5	−1.3 (3)	C11—C8—C9—C10	4.3 (3)
C2—C1—C6—N1	−179.85 (17)	C12—N3—C11—C8	−178.45 (16)
C4—C5—C6—C1	0.3 (3)	C7—C8—C11—N3	−173.72 (17)
C4—C5—C6—N1	178.91 (16)	C9—C8—C11—N3	4.1 (3)
C7—N1—C6—C1	−49.2 (3)	C11—N3—C12—C13	43.5 (3)
N2—N1—C6—C1	135.51 (17)	C11—N3—C12—C17	−140.12 (18)
C7—N1—C6—C5	132.20 (18)	C17—C12—C13—C14	0.1 (3)
N2—N1—C6—C5	−43.1 (2)	N3—C12—C13—C14	176.48 (18)
N2—N1—C7—C8	−0.27 (19)	C12—C13—C14—C15	0.5 (3)
C6—N1—C7—C8	−175.84 (15)	C13—C14—C15—C16	−0.2 (3)
N2—N1—C7—Cl1	177.06 (11)	C14—C15—C16—C17	−0.9 (3)
C6—N1—C7—Cl1	1.5 (2)	C15—C16—C17—C12	1.6 (3)
N1—C7—C8—C9	−0.30 (18)	C13—C12—C17—C16	−1.2 (3)
Cl1—C7—C8—C9	−177.38 (13)	N3—C12—C17—C16	−177.71 (18)

N1—C7—C8—C11	177.98 (15)

Fig. 1