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5-Chloro-4-[(2-chlorobenzoylhydrazono)-methyl]-3-methyl-1-phenyl-1*H*-pyrazole

Li-Rong Wen, Wen-Ying Qi, Zheng-Quan Zuo and Ming Li*

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, People's Republic of China

Correspondence e-mail: liming928@263.net

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Key indicators

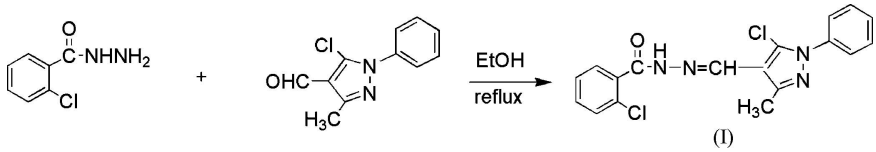
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.102
Data-to-parameter ratio = 13.9

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

In the title compound, $C_{18}H_{14}C_{12}N_4O$, centrosymmetrically related molecules are linked into dimers by intermolecular N—H···O hydrogen bonds. In the crystal structure, the molecules interact through intermolecular C—H···Cl hydrogen bonds, forming a three-dimensional network.

Comment

Pyrazole and its derivatives represent one of the most important classes of compounds, possessing a wide spectrum of biological activities, such as antibacterial, fungicidal, herbicidal and insecticidal activities (Iovu *et al.*, 2003). In the course of our systematic studies aimed at the synthesis of new bioactive compounds, we have synthesized the title compound, (I), and its structure is reported here.



(I)

The system defined by the chloromethylpyrazole ring and the C8/N2/N1/C7/O1 chain is roughly planar [maximum deviation 0.126 (2) Å for C7] and forms dihedral angles of 71.21 (5) and 45.14 (5)° with the C1–C6 and C13–C18 benzene rings, respectively. Bond distances and angles (Table 1) are as expected for this type of compound.

In the crystal structure, centrosymmetrically related molecules are linked into dimers by intermolecular N—H···O hydrogen bonds, thus generating graph-set motif $R_2^2(6)$ (Bernstein *et al.*, 1995). The dimers form a three-dimensional network *via* intermolecular C—H···Cl hydrogen bonds (Table 2 and Fig. 2).

Experimental

A mixture of 2-chlorobenzoylhydrazide (3 mmol, 0.512 g) and 5-chloro-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde (3 mmol,

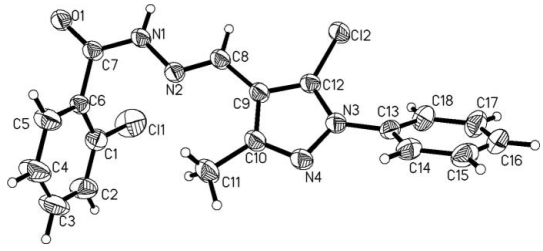


Figure 1
View of the title compound with 35% probability displacement ellipsoids.

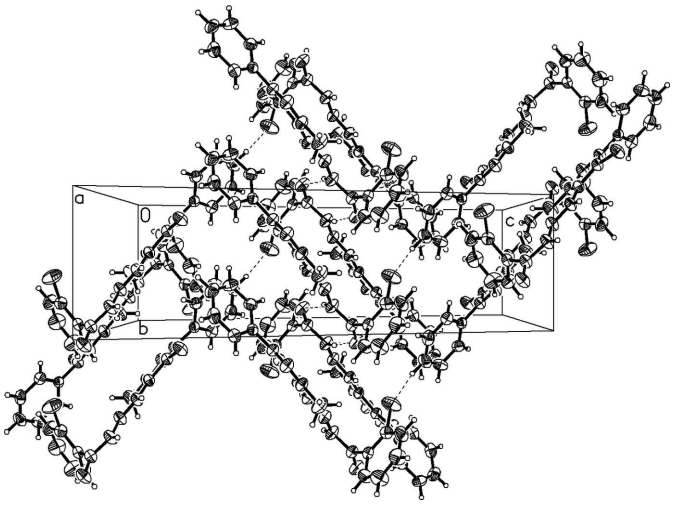


Figure 2
The molecular packing of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

0.662 g), synthesized according to the literature method of Wen *et al.*, 2005), was added to 15 ml ethanol and refluxed for 5 h. The solvent was removed under reduced pressure and the residue was recrystalized from ethanol. Single crystals suitable for X-ray analysis were obtained as colourless blocks by slow evaporation of the solvent (m.p. 452 K).

Crystal data

$C_{18}H_{14}Cl_2N_4O$
M_r = 373.23
Monoclinic, $C2/c$
a = 28.01 (2) Å
b = 6.199 (5) Å
c = 23.535 (18) Å
β = 119.199 (9)°
V = 3567 (5) Å^3
Z = 8
D_x = 1.390 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 293 (2) K
Block, colourless
0.32 × 0.28 × 0.26 mm

Data collection

Bruker APEX-II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.884, T_{max} = 0.907
9192 measured reflections
3145 independent reflections
2485 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.102
S = 1.09
3145 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 0.8269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.230 (2)	N3—N4	1.369 (2)
N1—C7	1.345 (2)	N4—C10	1.321 (2)
N2—C8	1.268 (2)	C9—C12	1.370 (3)
N3—C12	1.358 (2)	C9—C10	1.422 (3)
C12—N3—N4	110.04 (15)	N4—C10—C9	111.29 (15)
C10—N4—N3	106.04 (14)	N3—C12—C9	108.65 (14)
C12—C9—C10	103.98 (15)		
C7—N1—N2—C8	173.96 (17)	N1—N2—C8—C9	175.69 (16)
C12—N3—N4—C10	0.3 (2)	N2—C8—C9—C12	179.28 (18)
N2—N1—C7—O1	172.09 (17)	C12—N3—C13—C18	−47.3 (3)
N2—N1—C7—C6	−7.3 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.03	2.874 (3)	168
C16—H16···Cl1^{ii}	0.93	2.71	3.520 (4)	146

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

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å and N—H = 0.86 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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