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5-(2,4-Dichlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde

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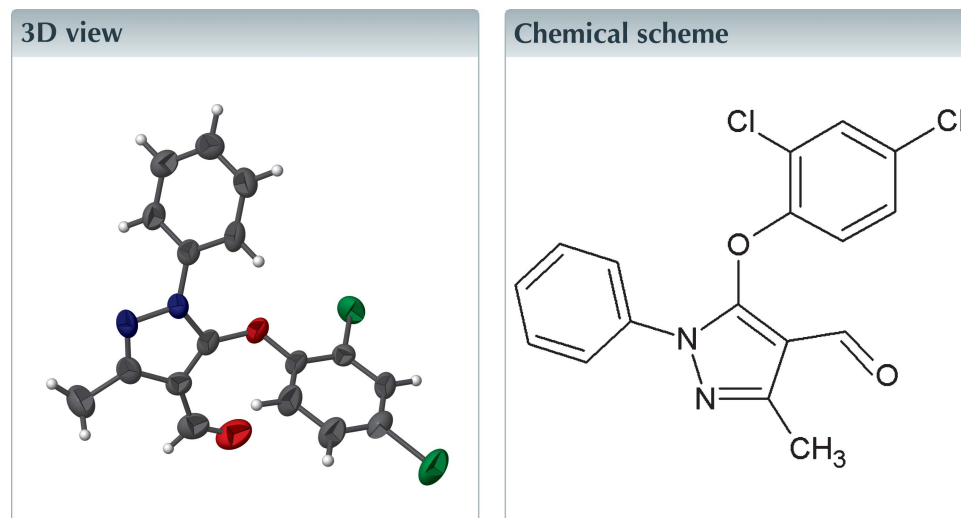
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Keywords: crystal structure; pyrazole; intermolecular hydrogen bonds.

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

In the crystal structure of the title compound, C₁₇H₁₂Cl₂N₂O₂, the pyrazole ring makes dihedral angles of 65.0 (2) and 43.9 (2)° with the dichlorophenyl and phenyl rings, respectively. The dihedral angle between the chlorophenyl and phenyl rings is 59.1 (2)°. In the crystal, the molecules are linked by C—H···O hydrogen bonds and weak C—Cl···π and C—H···π interactions, generating a three-dimensional network.



Structure description

As part of a research project on the synthesis and crystal structure determination of pyrazole derivatives, the structure of 5-(2,4-dichlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde is reported (Fig. 1).

The pyrazole (C1–C3/N1/N2) ring makes dihedral angles of 65.0 (2) and 43.9 (2)° with the dichlorophenyl (C12–C17) and phenyl (C4–C9) rings, respectively. The chlorophenyl ring makes a dihedral angle of 59.1 (2)° with the phenyl ring. In the crystal (Fig. 2), molecules are connected *via* C8–H8···O2ⁱ hydrogen bonds (Table 1). In addition, weak C—H···π interactions are observed [C13–H13···Cg2ⁱⁱ, with H13···Cg2ⁱⁱ = 2.95 Å, and C15–Cl2···Cg1ⁱⁱⁱ, with Cl2···Cg1ⁱⁱⁱ = 3.582 (4) Å; Cg1 and Cg2 are the centroids of the C1–C3/N1/N2 and C4–C9 rings, respectively; symmetry codes: (ii) $-x + 1, -y - 1, -z + 1$; (iii) $x - \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$].

Synthesis and crystallization

5-Chloro-4-formyl-3-methyl-1-phenyl-1*H*-pyrazole (0.1 mmol) and 2,4-dichlorophenol (0.1 mmol) were dissolved in dimethyl sulfoxide in a round-bottomed flask and the

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C8-H8\cdots O2^i$	0.93	2.50	3.297 (6)	144

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

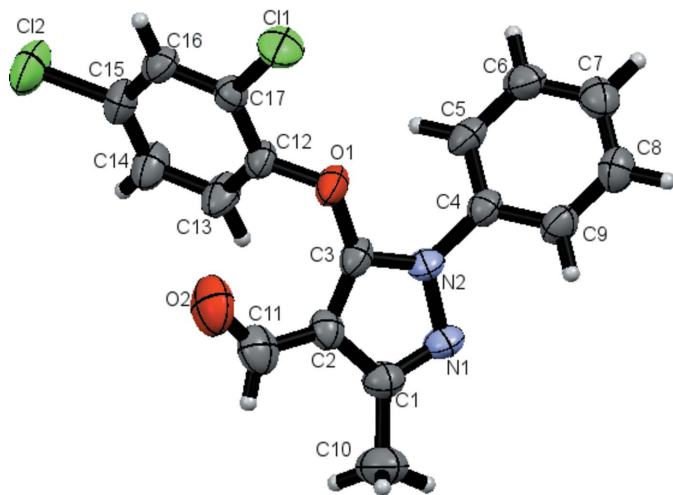


Figure 1
A view of the title molecule, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

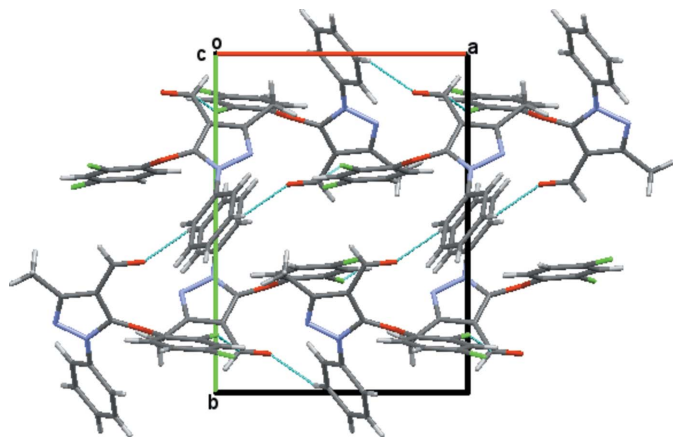


Figure 2
A view along the c axis of the crystal packing of the title compound. Hydrogen bonds are drawn as dashed lines.

solution refluxed for 4 h. After completion of the reaction, the reaction mixture was poured into crushed ice. The solid obtained was recrystallized from ethanol solution.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{12}Cl_2N_2O_2$
M_r	347.19
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (Å)	10.113 (8), 13.278 (10), 12.224 (10)
β (°)	102.219 (15)
V (Å ³)	1604 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.42
Crystal size (mm)	0.32 × 0.23 × 0.21
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Multi-scan (NUMABS; Rigaku 1999)
T_{min}, T_{max}	0.891, 0.916
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12776, 2911, 2127
R_{int}	0.059
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.072, 0.182, 1.21
No. of reflections	2911
No. of parameters	209
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.22, -0.20

Computer programs: *CrystalClear SM Expert* (Rigaku, 2011), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161111 [doi:10.1107/S2414314616011111]

5-(2,4-Dichlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde

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5-(2,4-Dichlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde*Crystal data*

$C_{17}H_{12}Cl_2N_2O_2$

$M_r = 347.19$

Monoclinic, $P2_1/n$

$a = 10.113$ (8) Å

$b = 13.278$ (10) Å

$c = 12.224$ (10) Å

$\beta = 102.219$ (15)°

$V = 1604$ (2) Å³

$Z = 4$

$F(000) = 712$

$D_x = 1.438$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å

Cell parameters from 2911 reflections

$\theta = 3.1$ – 25.3 °

$\mu = 0.42$ mm⁻¹

$T = 293$ K

Block, brown

$0.32 \times 0.23 \times 0.21$ mm

Data collection

Rigaku Saturn724+

diffractometer

profile data from ω -scans

Absorption correction: multi-scan

(NUMABS; Rigaku 1999)

$T_{\min} = 0.891$, $T_{\max} = 0.916$

12776 measured reflections

2911 independent reflections

2127 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.059$

$\theta_{\max} = 25.3$ °, $\theta_{\min} = 3.1$ °

$h = -12 \rightarrow 12$

$k = -15 \rightarrow 15$

$l = -14 \rightarrow 14$

2911 standard reflections

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.072$

$wR(F^2) = 0.182$

$S = 1.21$

2911 reflections

209 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0764P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.22$ e Å⁻³

$\Delta\rho_{\min} = -0.20$ e Å⁻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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	−0.00103 (11)	0.17058 (9)	0.30659 (9)	0.0662 (4)
C12	−0.06627 (12)	0.10625 (11)	0.72543 (11)	0.0819 (5)
O1	0.2843 (2)	0.1852 (2)	0.4076 (2)	0.0520 (8)
O2	0.2891 (4)	0.3853 (3)	0.5424 (3)	0.0861 (11)
N1	0.6373 (3)	0.2049 (3)	0.4479 (3)	0.0514 (9)
N2	0.5120 (3)	0.1601 (2)	0.4098 (3)	0.0442 (8)
C1	0.6133 (4)	0.2838 (3)	0.5059 (3)	0.0506 (10)
C2	0.4743 (4)	0.2941 (3)	0.5058 (3)	0.0459 (10)
C3	0.4154 (3)	0.2133 (3)	0.4444 (3)	0.0439 (10)
C4	0.4995 (3)	0.0817 (3)	0.3285 (3)	0.0428 (9)
C5	0.4137 (3)	0.0008 (3)	0.3318 (3)	0.0500 (10)
H5	0.3665	−0.0048	0.3891	0.060*
C6	0.3992 (4)	−0.0712 (3)	0.2491 (4)	0.0563 (11)
H6	0.3414	−0.1254	0.2502	0.068*
C7	0.4700 (4)	−0.0635 (4)	0.1646 (4)	0.0579 (11)
H7	0.4590	−0.1120	0.1085	0.070*
C8	0.5571 (4)	0.0161 (3)	0.1633 (3)	0.0540 (11)
H8	0.6058	0.0208	0.1069	0.065*
C9	0.5721 (4)	0.0887 (3)	0.2453 (3)	0.0496 (10)
H9	0.6311	0.1424	0.2446	0.060*
C10	0.7282 (4)	0.3492 (4)	0.5623 (4)	0.0782 (15)
H10A	0.8122	0.3203	0.5532	0.117*
H10B	0.7279	0.3543	0.6406	0.117*
H10C	0.7182	0.4151	0.5292	0.117*
C11	0.4077 (5)	0.3774 (4)	0.5475 (3)	0.0631 (12)
H11	0.4624	0.4297	0.5816	0.076*
C12	0.2070 (3)	0.1646 (3)	0.4873 (3)	0.0423 (9)
C13	0.2603 (4)	0.1510 (3)	0.5986 (3)	0.0590 (12)
H13	0.3535	0.1543	0.6250	0.071*
C14	0.1764 (4)	0.1322 (4)	0.6723 (4)	0.0595 (12)
H14	0.2128	0.1238	0.7483	0.071*
C15	0.0396 (4)	0.1260 (3)	0.6325 (4)	0.0506 (10)
C16	−0.0160 (4)	0.1374 (3)	0.5201 (3)	0.0477 (10)
H16	−0.1090	0.1326	0.4936	0.057*
C17	0.0686 (4)	0.1561 (3)	0.4475 (3)	0.0403 (9)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0528 (7)	0.0950 (9)	0.0438 (6)	−0.0045 (6)	−0.0053 (5)	−0.0044 (6)
C12	0.0684 (8)	0.1111 (11)	0.0782 (9)	−0.0020 (7)	0.0425 (7)	0.0116 (7)
O1	0.0324 (14)	0.086 (2)	0.0388 (15)	0.0022 (13)	0.0103 (12)	−0.0021 (14)
O2	0.078 (2)	0.101 (3)	0.082 (3)	0.036 (2)	0.022 (2)	−0.0045 (19)
N1	0.0301 (17)	0.062 (2)	0.059 (2)	−0.0052 (16)	0.0033 (15)	−0.0077 (18)
N2	0.0282 (16)	0.056 (2)	0.0470 (19)	−0.0018 (14)	0.0037 (14)	−0.0067 (16)

C1	0.045 (2)	0.055 (3)	0.049 (2)	-0.003 (2)	0.003 (2)	0.000 (2)
C2	0.046 (2)	0.055 (2)	0.036 (2)	0.006 (2)	0.0070 (18)	-0.0007 (19)
C3	0.0322 (19)	0.061 (3)	0.039 (2)	0.0055 (19)	0.0094 (17)	0.006 (2)
C4	0.0347 (19)	0.050 (2)	0.044 (2)	0.0021 (18)	0.0087 (18)	0.0028 (19)
C5	0.032 (2)	0.064 (3)	0.056 (3)	-0.0037 (19)	0.0126 (19)	0.008 (2)
C6	0.041 (2)	0.054 (3)	0.073 (3)	-0.006 (2)	0.010 (2)	-0.009 (2)
C7	0.050 (2)	0.069 (3)	0.051 (3)	0.006 (2)	0.005 (2)	-0.008 (2)
C8	0.054 (2)	0.063 (3)	0.049 (3)	0.006 (2)	0.018 (2)	0.006 (2)
C9	0.046 (2)	0.051 (2)	0.056 (3)	-0.0064 (19)	0.020 (2)	0.004 (2)
C10	0.063 (3)	0.075 (3)	0.093 (4)	-0.015 (2)	0.008 (3)	-0.016 (3)
C11	0.073 (3)	0.075 (3)	0.040 (2)	0.009 (3)	0.010 (2)	0.004 (2)
C12	0.0315 (19)	0.055 (2)	0.042 (2)	0.0054 (17)	0.0122 (17)	-0.0004 (19)
C13	0.032 (2)	0.095 (3)	0.048 (3)	0.006 (2)	0.0052 (19)	0.009 (2)
C14	0.046 (2)	0.088 (3)	0.046 (2)	0.008 (2)	0.014 (2)	0.012 (2)
C15	0.048 (2)	0.056 (3)	0.054 (3)	0.007 (2)	0.025 (2)	0.006 (2)
C16	0.032 (2)	0.051 (2)	0.062 (3)	0.0007 (18)	0.013 (2)	-0.004 (2)
C17	0.038 (2)	0.042 (2)	0.040 (2)	0.0042 (17)	0.0074 (17)	-0.0027 (17)

Geometric parameters (Å, °)

C11—C17	1.728 (4)	C7—H7	0.9300
C12—C15	1.737 (4)	C7—C8	1.378 (6)
O1—C3	1.359 (4)	C8—H8	0.9300
O1—C12	1.399 (4)	C8—C9	1.376 (6)
O2—C11	1.193 (5)	C9—H9	0.9300
N1—N2	1.387 (4)	C10—H10A	0.9600
N1—C1	1.316 (5)	C10—H10B	0.9600
N2—C3	1.344 (4)	C10—H10C	0.9600
N2—C4	1.426 (5)	C11—H11	0.9300
C1—C2	1.411 (5)	C12—C13	1.366 (5)
C1—C10	1.498 (6)	C12—C17	1.385 (5)
C2—C3	1.371 (5)	C13—H13	0.9300
C2—C11	1.443 (6)	C13—C14	1.385 (5)
C4—C5	1.387 (5)	C14—H14	0.9300
C4—C9	1.378 (5)	C14—C15	1.369 (6)
C5—H5	0.9300	C15—C16	1.379 (6)
C5—C6	1.376 (6)	C16—H16	0.9300
C6—H6	0.9300	C16—C17	1.380 (5)
C6—C7	1.379 (6)		
C3—O1—C12	118.2 (3)	C8—C9—C4	119.8 (4)
C1—N1—N2	105.2 (3)	C8—C9—H9	120.1
N1—N2—C4	119.1 (3)	C1—C10—H10A	109.5
C3—N2—N1	110.0 (3)	C1—C10—H10B	109.5
C3—N2—C4	129.8 (3)	C1—C10—H10C	109.5
N1—C1—C2	112.0 (3)	H10A—C10—H10B	109.5
N1—C1—C10	119.8 (4)	H10A—C10—H10C	109.5
C2—C1—C10	128.3 (4)	H10B—C10—H10C	109.5

C1—C2—C11	127.8 (4)	O2—C11—C2	126.1 (5)
C3—C2—C1	104.0 (3)	O2—C11—H11	117.0
C3—C2—C11	127.7 (4)	C2—C11—H11	117.0
O1—C3—C2	132.6 (3)	C13—C12—O1	124.0 (3)
N2—C3—O1	118.3 (3)	C13—C12—C17	119.7 (3)
N2—C3—C2	108.8 (3)	C17—C12—O1	116.3 (3)
C5—C4—N2	120.6 (3)	C12—C13—H13	119.8
C9—C4—N2	118.8 (3)	C12—C13—C14	120.3 (4)
C9—C4—C5	120.5 (4)	C14—C13—H13	119.8
C4—C5—H5	120.4	C13—C14—H14	120.3
C6—C5—C4	119.1 (4)	C15—C14—C13	119.5 (4)
C6—C5—H5	120.4	C15—C14—H14	120.3
C5—C6—H6	119.8	C14—C15—C12	119.5 (3)
C5—C6—C7	120.5 (4)	C14—C15—C16	121.0 (4)
C7—C6—H6	119.8	C16—C15—C12	119.4 (3)
C6—C7—H7	120.0	C15—C16—H16	120.5
C8—C7—C6	120.0 (4)	C15—C16—C17	118.9 (3)
C8—C7—H7	120.0	C17—C16—H16	120.5
C7—C8—H8	119.9	C12—C17—C11	120.6 (3)
C9—C8—C7	120.1 (4)	C16—C17—C11	118.9 (3)
C9—C8—H8	119.9	C16—C17—C12	120.5 (4)
C4—C9—H9	120.1		
C12—C15—C16—C17	178.1 (3)	C4—N2—C3—O1	7.3 (6)
O1—C12—C13—C14	179.1 (4)	C4—N2—C3—C2	-167.6 (4)
O1—C12—C17—C11	0.0 (5)	C4—C5—C6—C7	0.5 (6)
O1—C12—C17—C16	-179.0 (3)	C5—C4—C9—C8	1.5 (6)
N1—N2—C3—O1	174.9 (3)	C5—C6—C7—C8	0.7 (6)
N1—N2—C3—C2	0.0 (4)	C6—C7—C8—C9	-0.9 (6)
N1—N2—C4—C5	143.8 (4)	C7—C8—C9—C4	-0.2 (6)
N1—N2—C4—C9	-37.6 (5)	C9—C4—C5—C6	-1.6 (6)
N1—C1—C2—C3	1.2 (4)	C10—C1—C2—C3	-178.6 (4)
N1—C1—C2—C11	-171.3 (4)	C10—C1—C2—C11	8.9 (7)
N2—N1—C1—C2	-1.1 (4)	C11—C2—C3—O1	-2.0 (7)
N2—N1—C1—C10	178.6 (4)	C11—C2—C3—N2	171.8 (4)
N2—C4—C5—C6	177.0 (3)	C12—O1—C3—N2	124.8 (3)
N2—C4—C9—C8	-177.2 (3)	C12—O1—C3—C2	-61.9 (5)
C1—N1—N2—C3	0.7 (4)	C12—C13—C14—C15	0.9 (7)
C1—N1—N2—C4	169.8 (3)	C13—C12—C17—C11	-178.8 (3)
C1—C2—C3—O1	-174.5 (4)	C13—C12—C17—C16	2.3 (6)
C1—C2—C3—N2	-0.7 (4)	C13—C14—C15—C12	-178.0 (3)
C1—C2—C11—O2	177.0 (4)	C13—C14—C15—C16	0.5 (7)
C3—O1—C12—C13	-13.8 (5)	C14—C15—C16—C17	-0.5 (6)
C3—O1—C12—C17	167.5 (3)	C15—C16—C17—C11	-179.9 (3)
C3—N2—C4—C5	-49.5 (6)	C15—C16—C17—C12	-0.9 (5)
C3—N2—C4—C9	129.1 (4)	C17—C12—C13—C14	-2.2 (6)
C3—C2—C11—O2	6.3 (7)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C8—H8···O2 ⁱ	0.93	2.50	3.297 (6)	144

Symmetry code: (i) $x+1/2, -y+1/2, z-1/2$.