IUCrData

ISSN 2414-3146

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

S. Madan Kumar,[a] N. Manju,[b] Asma,[b] Balakrishna Kalluraya,[b] K. Byrappa[c] and Ismail Warad[d]*

[a]PURSE Lab, Mangalagangotri, Mangalore University, Mangaluru 574 199, India, [b]Department of Post-Graduate and Research In Chemistry, Mangalagangori, Mangalore University, India, [c]Department of Material Science, Mangalore University, Mangaluru 574 199, India, and [d]Department of Chemistry, Faculty of Science, An-Najah National University, Nablus, West Bank, Palestinian Territories. *Correspondence e-mail: khalil.i@najah.edu

Received 6 July 2016
Accepted 8 July 2016

Edited by L. Van Meervelt, Katholieke Universiteit Leuven, Belgium

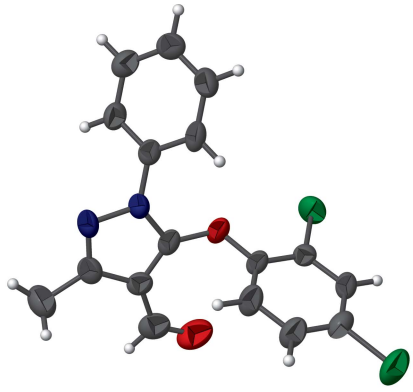
Keywords: crystal structure; pyrazole; intermolecular hydrogen bonds.

CCDC reference: 1491572

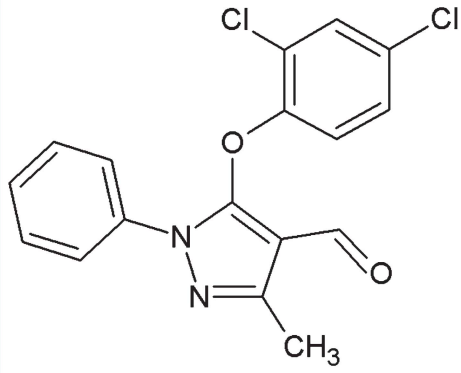
Structural data: full structural data are available from iucrdata.iucr.org

In the crystal structure of the title compound, $C_{17}H_{12}Cl_2N_2O_2$, 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.

3D view



Chemical scheme



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···O2^{i} hydrogen bonds (Table 1). In addition, weak C—H···π interactions are observed [C13—H13···*Cg*2^{ii}, with H13···*Cg*2^{ii} = 2.95 Å, and C15—Cl2···*Cg*1^{iii}, with Cl2···*Cg*1^{iii} = 3.582 (4) Å; *Cg*1 and *Cg*2 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

OPEN ACCESS

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2^{i}	0.93	2.50	3.297 (6)	144

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

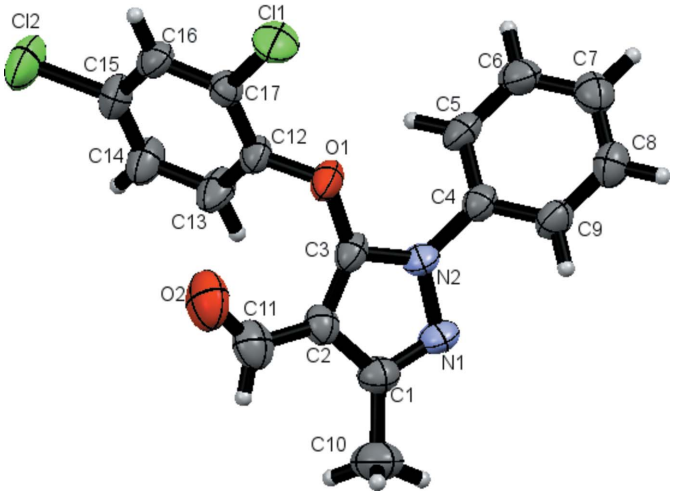


Figure 1
A view of the title molecule, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

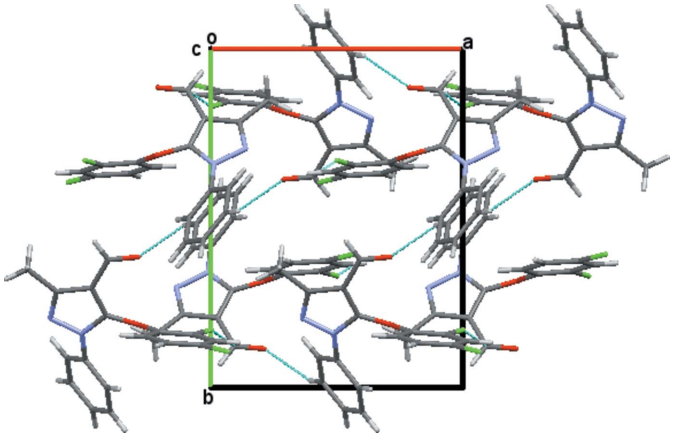


Figure 2
A view along the c axis of the crystal packing of the title compound. Hydrogen bonds are drawn as dashed lines.

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

Authors thank DST–PURSE, Mangalore University, Mangaluru, for providing the single-crystal X-ray diffraction facility.

References

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Rigaku (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2011). *CrystalClear SM Expert*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

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

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

S. Madan Kumar, N. Manju, Asma, Balakrishna Kalluraya, K. Byrappa and Ismail Warad

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
β = 102.219 (15)°
V = 1604 (2) Å^3
Z = 4
$F(000)$ = 712
D_x = 1.438 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 2911 reflections
θ = 3.1–25.3°
μ = 0.42 mm^{-1}
T = 293 K
Block, brown
0.32 × 0.23 × 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_{int} = 0.059
θ_{max} = 25.3°, θ_{min} = 3.1°
h = −12→12
k = −15→15
l = −14→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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.00103 (11)	0.17058 (9)	0.30659 (9)	0.0662 (4)
Cl2	−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 (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0528 (7)	0.0950 (9)	0.0438 (6)	−0.0045 (6)	−0.0053 (5)	−0.0044 (6)
Cl2	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, º)

Cl1—C17	1.728 (4)	C7—H7	0.9300
Cl2—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—Cl2	119.5 (3)
C5—C6—C7	120.5 (4)	C14—C15—C16	121.0 (4)
C7—C6—H6	119.8	C16—C15—Cl2	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—Cl1	120.6 (3)
C9—C8—C7	120.1 (4)	C16—C17—Cl1	118.9 (3)
C9—C8—H8	119.9	C16—C17—C12	120.5 (4)
C4—C9—H9	120.1		

Cl2—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—Cl1	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—Cl1	−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—Cl2	−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—Cl1	−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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2^{i}	0.93	2.50	3.297 (6)	144

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