Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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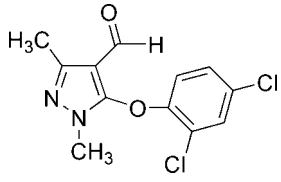
Received 28 October 2011; accepted 31 October 2011

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.054; wR factor = 0.146; data-to-parameter ratio = 17.9.

In the title molecule, $C_{12}H_{10}Cl_2N_2O_2$, the benzene and pyrazole rings form a dihedral angle of 72.8 (3)°. In the crystal, weak intermolecular C—H···O hydrogen bonds link the molecules into chains along [01$\bar{1}$].

Related literature

For the crystal structure of a related pyrazole derivative studied recently by our group, see: Shen *et al.* (2011).



Experimental

Crystal data

$C_{12}H_{10}Cl_2N_2O_2$
M_r = 285.12
Triclinic, $P\bar{1}$
a = 8.018 (3) Å
b = 8.505 (3) Å
c = 10.365 (5) Å
α = 74.56 (2)°
β = 83.96 (3)°
γ = 67.30 (2)°
V = 628.5 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.51 mm^{-1}
T = 113 K
0.24 × 0.22 × 0.20 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2008)
T_{min} = 0.887, T_{max} = 0.905
6391 measured reflections
2948 independent reflections
2766 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.146
S = 1.06
2948 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O2^{i}	0.95	2.45	3.284 (3)	147
C10—H10C···O2^{ii}	0.98	2.46	3.391 (3)	158

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+2, -y, -z+2$.

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

This work was supported by the Scientific Research Foundation for Talent Introduction of Nantong University (grant No. 03080226).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5188).

References

Rigaku (2008). *CrystalClear*. Rigaku Corporation, Toyko, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shen, Y.-J., Xu, M. & Fan, C.-G. (2011). *Acta Cryst.* E**67**, o2936.

supporting information

Acta Cryst. (2011). E**67**, o3194 [https://doi.org/10.1107/S1600536811045831]

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

Yong-Jun Shen, Mei Xu and Chong-Guang Fan

S1. Comment

As a continuation of our structural study of pyrazole derivatives (Shen *et al.*, 2011), we present here the title compound (I).

In (I) (Fig. 1), all bond lengths and angles are normal and correspond to those observed in the related compound (Shen *et al.*, 2011). The dihedral angle between the substituted benzene ring and pyrazole ring is 72.8 (3) °. The crystal packing exhibits weak intermolecular C—H···O interactions (Table 1), which link molecules into chains along [01-1].

S2. Experimental

To a stirred solution of 1-methyl-3-methyl-5-chloro-1*H*-pyrazole- 4-carbaldehyde(30 mmol) and 2,4-dichlorophenol(48 mmol) in DMF(30 ml) was added potassium hydroxide(60 mmol) at room temperature. The resulting mixture was heated to 388 k for 6 h. Then the reaction solution was poured into cold water(100 ml) and extracted with ethyl acetate (3 * 60 ml). The organic layer was dried over anhydrous magnesium sulfate. After removal of the solvent, the residue was recrystallized from ethyl acetate/petroleum ether to give colourless crystals.

S3. Refinement

All H atoms were placed in calculated positions, with C–H = 0.95 - 0.98 ° A, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2-1.5 U_{eq}(C).

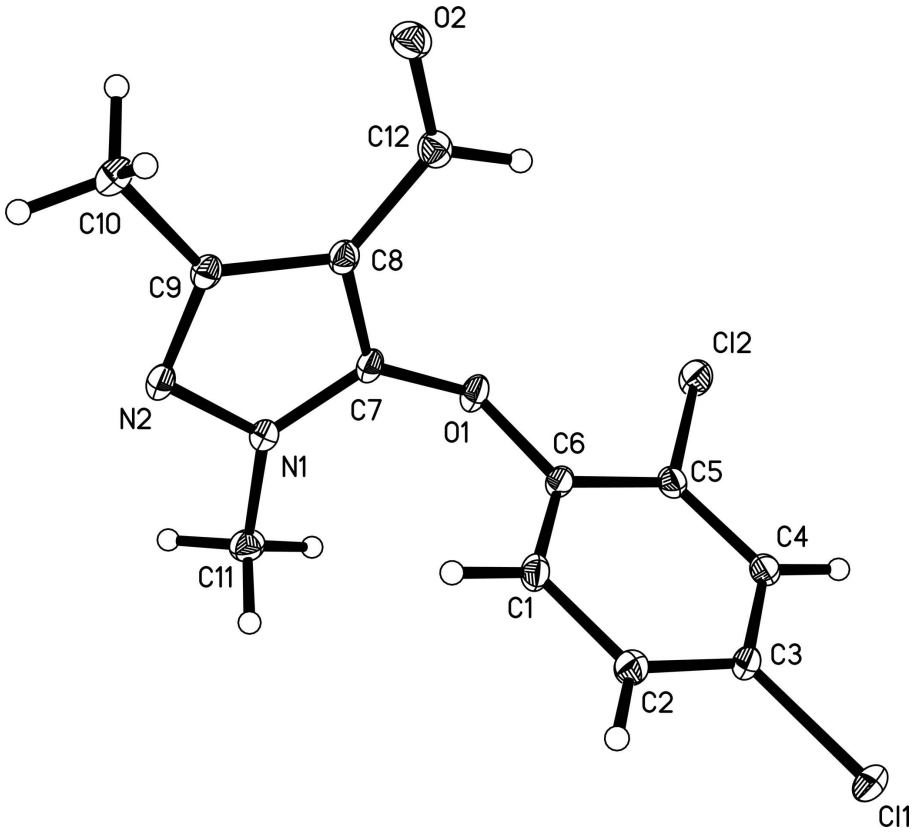


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

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

Crystal data

$C_{12}H_{10}Cl_2N_2O_2$
$M_r = 285.12$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.018$ (3) Å
$b = 8.505$ (3) Å
$c = 10.365$ (5) Å
$\alpha = 74.56$ (2)°
$\beta = 83.96$ (3)°
$\gamma = 67.30$ (2)°
$V = 628.5$ (5) Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.507$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2204 reflections
$\theta = 2.0$–$27.9°$
$\mu = 0.51$ mm^{-1}
$T = 113$ K
Prism, colourless
0.24 × 0.22 × 0.20 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.22 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2008)
$T_{min} = 0.887$, $T_{max} = 0.905$
6391 measured reflections
2948 independent reflections
2766 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.0°$
$h = -10 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.146$
$S = 1.06$
2948 reflections
165 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0863P)^2 + 0.4717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.32121 (7)	0.81388 (7)	0.29679 (5)	0.03004 (17)
Cl2	0.90839 (7)	0.84252 (7)	0.48228 (5)	0.02853 (17)
O1	0.7986 (2)	0.68007 (19)	0.73949 (13)	0.0229 (3)
O2	1.1787 (2)	0.15447 (19)	0.88478 (15)	0.0268 (3)
N1	0.6770 (2)	0.6174 (2)	0.95613 (15)	0.0200 (3)
N2	0.7105 (2)	0.4841 (2)	1.07097 (15)	0.0203 (3)
C1	0.5338 (3)	0.6535 (3)	0.66259 (18)	0.0220 (4)
H1	0.5090	0.5963	0.7502	0.026*
C2	0.4222 (3)	0.6874 (3)	0.55656 (19)	0.0233 (4)
H2	0.3203	0.6540	0.5711	0.028*
C3	0.4611 (3)	0.7702 (3)	0.42976 (19)	0.0225 (4)
C4	0.6096 (3)	0.8200 (2)	0.40452 (19)	0.0220 (4)
H4	0.6347	0.8762	0.3167	0.026*
C5	0.7201 (3)	0.7854 (2)	0.51096 (18)	0.0201 (4)
C6	0.6823 (3)	0.7040 (2)	0.63958 (18)	0.0194 (4)
C7	0.7948 (3)	0.5673 (2)	0.85978 (17)	0.0186 (4)
C8	0.9135 (3)	0.3965 (2)	0.90937 (18)	0.0194 (4)
C9	0.8528 (3)	0.3516 (2)	1.04350 (18)	0.0188 (4)
C10	0.9292 (3)	0.1822 (3)	1.1457 (2)	0.0249 (4)
H10A	0.8626	0.1912	1.2300	0.037*
H10B	1.0569	0.1563	1.1602	0.037*
H10C	0.9185	0.0875	1.1143	0.037*
C11	0.5330 (3)	0.7878 (3)	0.9522 (2)	0.0245 (4)
H11A	0.5564	0.8774	0.8794	0.037*
H11B	0.5286	0.8182	1.0376	0.037*

H11C	0.4170	0.7822	0.9369	0.037*
C12	1.0679 (3)	0.3008 (3)	0.8386 (2)	0.0226 (4)
H12	1.0848	0.3566	0.7485	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0258 (3)	0.0415 (3)	0.0194 (3)	−0.0095 (2)	−0.00688 (19)	−0.0039 (2)
Cl2	0.0265 (3)	0.0386 (3)	0.0221 (3)	−0.0183 (2)	0.00085 (19)	−0.0012 (2)
O1	0.0273 (7)	0.0298 (7)	0.0143 (6)	−0.0164 (6)	−0.0027 (5)	0.0001 (5)
O2	0.0248 (7)	0.0256 (7)	0.0293 (7)	−0.0064 (6)	0.0004 (6)	−0.0104 (6)
N1	0.0201 (8)	0.0219 (8)	0.0159 (7)	−0.0060 (6)	−0.0012 (6)	−0.0034 (6)
N2	0.0240 (8)	0.0236 (8)	0.0142 (7)	−0.0107 (7)	−0.0025 (6)	−0.0022 (6)
C1	0.0255 (10)	0.0260 (9)	0.0141 (8)	−0.0108 (8)	0.0007 (7)	−0.0030 (7)
C2	0.0218 (9)	0.0284 (10)	0.0206 (9)	−0.0108 (8)	−0.0001 (7)	−0.0051 (7)
C3	0.0226 (9)	0.0262 (9)	0.0149 (8)	−0.0045 (8)	−0.0027 (7)	−0.0049 (7)
C4	0.0207 (9)	0.0216 (9)	0.0175 (8)	−0.0027 (7)	0.0014 (7)	−0.0032 (7)
C5	0.0199 (9)	0.0215 (9)	0.0169 (8)	−0.0069 (7)	0.0029 (7)	−0.0039 (7)
C6	0.0207 (9)	0.0202 (8)	0.0151 (8)	−0.0048 (7)	−0.0024 (7)	−0.0041 (6)
C7	0.0218 (9)	0.0231 (9)	0.0131 (7)	−0.0110 (7)	−0.0026 (6)	−0.0030 (6)
C8	0.0204 (9)	0.0232 (9)	0.0165 (8)	−0.0100 (7)	−0.0016 (7)	−0.0049 (7)
C9	0.0207 (9)	0.0210 (8)	0.0165 (8)	−0.0094 (7)	−0.0026 (6)	−0.0041 (6)
C10	0.0272 (10)	0.0230 (9)	0.0216 (9)	−0.0087 (8)	−0.0062 (7)	0.0005 (7)
C11	0.0232 (10)	0.0215 (9)	0.0254 (9)	−0.0038 (8)	−0.0032 (7)	−0.0061 (7)
C12	0.0235 (9)	0.0251 (9)	0.0222 (9)	−0.0101 (8)	0.0001 (7)	−0.0092 (7)

Geometric parameters (Å, º)

Cl1—C3	1.740 (2)	C4—C5	1.386 (3)
Cl2—C5	1.731 (2)	C4—H4	0.9500
O1—C7	1.361 (2)	C5—C6	1.391 (3)
O1—C6	1.388 (2)	C7—C8	1.385 (3)
O2—C12	1.219 (3)	C8—C9	1.426 (3)
N1—C7	1.338 (2)	C8—C12	1.443 (3)
N1—N2	1.374 (2)	C9—C10	1.489 (3)
N1—C11	1.456 (3)	C10—H10A	0.9800
N2—C9	1.328 (3)	C10—H10B	0.9800
C1—C2	1.390 (3)	C10—H10C	0.9800
C1—C6	1.392 (3)	C11—H11A	0.9800
C1—H1	0.9500	C11—H11B	0.9800
C2—C3	1.382 (3)	C11—H11C	0.9800
C2—H2	0.9500	C12—H12	0.9500
C3—C4	1.389 (3)		
C7—O1—C6	118.55 (15)	N1—C7—C8	108.81 (16)
C7—N1—N2	110.80 (16)	O1—C7—C8	128.76 (17)
C7—N1—C11	128.50 (17)	C7—C8—C9	103.53 (16)
N2—N1—C11	120.66 (16)	C7—C8—C12	125.35 (17)

C9—N2—N1	105.68 (15)	C9—C8—C12	130.95 (18)
C2—C1—C6	119.56 (17)	N2—C9—C8	111.17 (17)
C2—C1—H1	120.2	N2—C9—C10	121.08 (17)
C6—C1—H1	120.2	C8—C9—C10	127.74 (18)
C3—C2—C1	119.33 (19)	C9—C10—H10A	109.5
C3—C2—H2	120.3	C9—C10—H10B	109.5
C1—C2—H2	120.3	H10A—C10—H10B	109.5
C2—C3—C4	121.98 (18)	C9—C10—H10C	109.5
C2—C3—Cl1	119.51 (16)	H10A—C10—H10C	109.5
C4—C3—Cl1	118.50 (15)	H10B—C10—H10C	109.5
C5—C4—C3	118.21 (17)	N1—C11—H11A	109.5
C5—C4—H4	120.9	N1—C11—H11B	109.5
C3—C4—H4	120.9	H11A—C11—H11B	109.5
C4—C5—C6	120.74 (18)	N1—C11—H11C	109.5
C4—C5—Cl2	119.32 (14)	H11A—C11—H11C	109.5
C6—C5—Cl2	119.94 (15)	H11B—C11—H11C	109.5
O1—C6—C5	116.08 (17)	O2—C12—C8	125.49 (19)
O1—C6—C1	123.75 (16)	O2—C12—H12	117.3
C5—C6—C1	120.17 (17)	C8—C12—H12	117.3
N1—C7—O1	122.15 (17)		

C7—N1—N2—C9	0.7 (2)	C11—N1—C7—O1	2.7 (3)
C11—N1—N2—C9	−177.30 (16)	N2—N1—C7—C8	−0.6 (2)
C6—C1—C2—C3	0.2 (3)	C11—N1—C7—C8	177.13 (18)
C1—C2—C3—C4	0.4 (3)	C6—O1—C7—N1	−85.0 (2)
C1—C2—C3—Cl1	−179.69 (15)	C6—O1—C7—C8	101.7 (2)
C2—C3—C4—C5	−0.3 (3)	N1—C7—C8—C9	0.3 (2)
Cl1—C3—C4—C5	179.75 (14)	O1—C7—C8—C9	174.29 (17)
C3—C4—C5—C6	−0.4 (3)	N1—C7—C8—C12	−175.30 (17)
C3—C4—C5—Cl2	178.92 (14)	O1—C7—C8—C12	−1.3 (3)
C7—O1—C6—C5	−165.15 (17)	N1—N2—C9—C8	−0.4 (2)
C7—O1—C6—C1	15.6 (3)	N1—N2—C9—C10	179.92 (16)
C4—C5—C6—O1	−178.22 (16)	C7—C8—C9—N2	0.1 (2)
Cl2—C5—C6—O1	2.5 (2)	C12—C8—C9—N2	175.37 (19)
C4—C5—C6—C1	1.0 (3)	C7—C8—C9—C10	179.69 (18)
Cl2—C5—C6—C1	−178.27 (15)	C12—C8—C9—C10	−5.0 (3)
C2—C1—C6—O1	178.23 (17)	C7—C8—C12—O2	175.37 (19)
C2—C1—C6—C5	−0.9 (3)	C9—C8—C12—O2	1.0 (3)
N2—N1—C7—O1	−175.06 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.95	2.45	3.284 (3)	147
C10—H10*C*···O2^{ii}	0.98	2.46	3.391 (3)	158

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