

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

5-(2-Chlorophenoxy)-1,3-dimethyl-1H-pyrazole-4-carbaldehyde oxime

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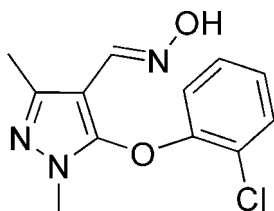
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Received 6 June 2012; accepted 12 June 2012

 Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.061; wR factor = 0.159; data-to-parameter ratio = 13.8.

In the title molecule, $\text{C}_{12}\text{H}_{12}\text{ClN}_3\text{O}_2$, the benzene and pyrazole rings are inclined to each other at a dihedral angle of $83.3(3)^\circ$. In the crystal, molecules are linked into [010] chains *via* $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds with the unsubstituted pyrazole N atom acting as the acceptor.

Related literature

 For a related structure, see: Dai *et al.* (2011).


Experimental

Crystal data

 $\text{C}_{12}\text{H}_{12}\text{ClN}_3\text{O}_2$
 $M_r = 265.70$

 Monoclinic, $P2_1/c$
 $a = 11.108(2)$ Å
 $b = 14.998(3)$ Å
 $c = 8.0839(16)$ Å
 $\beta = 104.94(3)^\circ$
 $V = 1301.2(4)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 113$ K
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

 Rigaku SCXmini diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2008)
 $T_{\min} = 0.918$, $T_{\max} = 0.944$

 10731 measured reflections
 2288 independent reflections
 1638 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.159$
 $S = 0.99$
 2288 reflections

 166 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{O2}-\text{H2}\cdots\text{N2}^i$ | 0.82 | 1.97 | 2.787 (3) | 171 |

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

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 Science Foundation of Nantong University (grant No. 11Z046).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5311).

References

- Dai, H., Miao, W.-K., Wu, S.-S., Qin, X. & Fang, J.-X. (2011). *Acta Cryst.* **E67**, o775.
 Rigaku (2008). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
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supplementary materials

Acta Cryst. (2012). E68, o2129 [doi:10.1107/S1600536812026530]

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

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Comment

In a continuation of our search for new pyrazole oxime derivatives (Dai *et al.*, 2011), we present here the title compound, (I).

In (I) (Fig. 1), all bond lengths and angles are normal and comparable with those observed in the related compound (Dai *et al.*, 2011). The plane of substituted phenyl ring makes a dihedral angle of 83.3 (3)° with the pyrazole ring. In the crystal, intermolecular O—H···N hydrogen bonds (Table1) link molecules into chains in [010].

Experimental

To a stirred solution of hydroxylamine hydrochloride (6 mmol) and potassium hydroxide (8 mmol) in methanol (30 ml) was added 5-(2-chlorophenoxy)-1,3-dimethyl-1*H*-pyrazole-4-carbaldehyde (4 mmol). The resulting mixture was heated to reflux for 6 h. The reaction mixture was cooled and poured into cold water (80 ml). The resulting colourless solid was collected and then recrystallized from ethyl acetate to give colourless crystals.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 and 0.96 ° Å, and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C})$.

Computing details

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

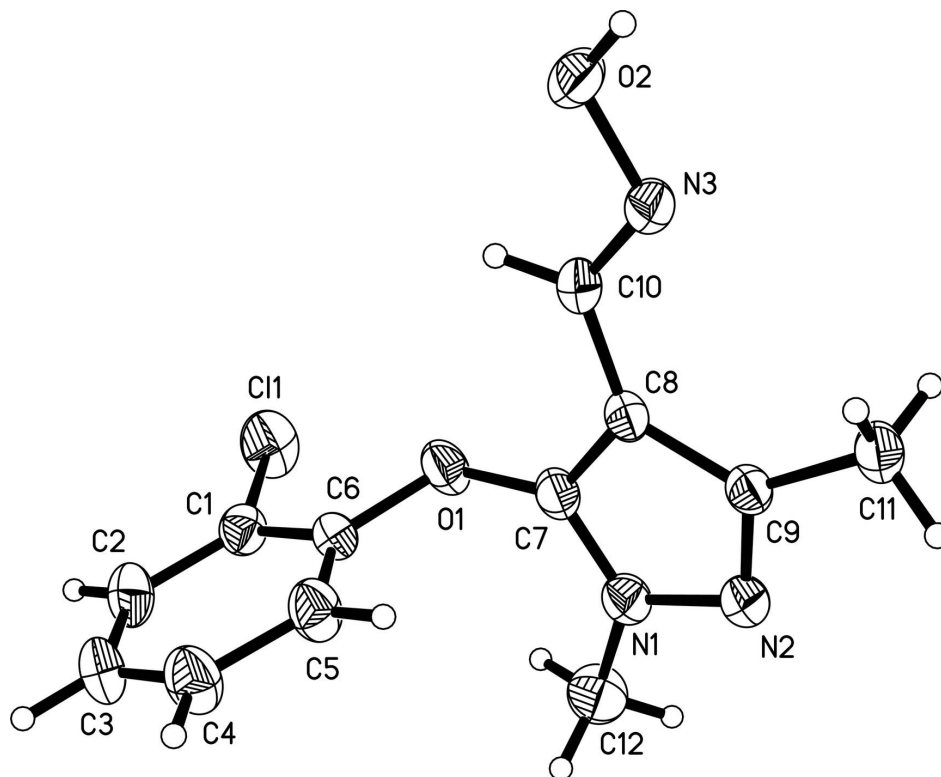


Figure 1

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

5-(2-Chlorophenoxy)-1,3-dimethyl-1H-pyrazole-4-carbaldehyde oxime

Crystal data

$C_{12}H_{12}ClN_3O_2$

$M_r = 265.70$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 11.108\ (2)\ \text{\AA}$

$b = 14.998\ (3)\ \text{\AA}$

$c = 8.0839\ (16)\ \text{\AA}$

$\beta = 104.94\ (3)^\circ$

$V = 1301.2\ (4)\ \text{\AA}^3$

$Z = 4$

$F(000) = 552$

$D_x = 1.356\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 10109 reflections

$\theta = 3.1\text{--}27.7^\circ$

$\mu = 0.29\ \text{mm}^{-1}$

$T = 113\ \text{K}$

Prism, colourless

$0.30 \times 0.25 \times 0.20\ \text{mm}$

Data collection

Rigaku SCXmini
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and φ scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2008)

$T_{\min} = 0.918$, $T_{\max} = 0.944$

10731 measured reflections

2288 independent reflections

1638 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.064$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.1^\circ$

$h = -13 \rightarrow 13$

$k = -17 \rightarrow 17$

$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.159$
 $S = 0.99$
 2288 reflections
 166 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
 $w = 1/[\sigma^2(F_o^2) + (0.0775P)^2 + 0.5684P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.096$
 $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|---------------|---------------|----------------------------------|
| Cl1 | 0.95727 (9) | 0.11946 (8) | -0.10002 (12) | 0.0797 (4) |
| O1 | 0.78065 (18) | 0.08065 (15) | 0.0915 (3) | 0.0555 (6) |
| N2 | 0.5175 (2) | 0.13782 (17) | 0.2347 (3) | 0.0504 (7) |
| N3 | 0.5840 (2) | -0.14771 (16) | 0.2182 (4) | 0.0526 (7) |
| N1 | 0.6218 (2) | 0.15655 (17) | 0.1805 (3) | 0.0502 (7) |
| C7 | 0.6784 (3) | 0.0805 (2) | 0.1570 (4) | 0.0459 (8) |
| C10 | 0.6468 (3) | -0.0833 (2) | 0.1825 (4) | 0.0489 (8) |
| H10 | 0.7180 | -0.0960 | 0.1466 | 0.059* |
| O2 | 0.6382 (2) | -0.22945 (15) | 0.1961 (4) | 0.0742 (8) |
| H2 | 0.5990 | -0.2700 | 0.2262 | 0.111* |
| C6 | 0.8951 (3) | 0.10481 (19) | 0.2006 (4) | 0.0437 (7) |
| C1 | 0.9878 (3) | 0.1260 (2) | 0.1212 (4) | 0.0488 (8) |
| C8 | 0.6126 (2) | 0.0091 (2) | 0.1949 (4) | 0.0418 (7) |
| C9 | 0.5111 (3) | 0.0494 (2) | 0.2418 (4) | 0.0421 (7) |
| C5 | 0.9187 (3) | 0.1079 (2) | 0.3747 (4) | 0.0585 (9) |
| H5 | 0.8565 | 0.0936 | 0.4281 | 0.070* |
| C2 | 1.1039 (3) | 0.1516 (2) | 0.2186 (5) | 0.0618 (10) |
| H2A | 1.1660 | 0.1663 | 0.1651 | 0.074* |
| C4 | 1.0363 (3) | 0.1327 (3) | 0.4714 (5) | 0.0685 (10) |
| H4 | 1.0533 | 0.1337 | 0.5903 | 0.082* |
| C11 | 0.4042 (3) | 0.0054 (2) | 0.2915 (4) | 0.0527 (8) |
| H11A | 0.3653 | 0.0475 | 0.3508 | 0.079* |
| H11B | 0.4344 | -0.0444 | 0.3650 | 0.079* |
| H11C | 0.3444 | -0.0150 | 0.1904 | 0.079* |
| C3 | 1.1281 (3) | 0.1556 (2) | 0.3924 (5) | 0.0649 (10) |
| H3 | 1.2060 | 0.1737 | 0.4574 | 0.078* |

| | | | | |
|------|------------|------------|------------|-------------|
| C12 | 0.6535 (4) | 0.2479 (2) | 0.1475 (5) | 0.0698 (11) |
| H12A | 0.7002 | 0.2747 | 0.2523 | 0.105* |
| H12B | 0.5785 | 0.2813 | 0.1026 | 0.105* |
| H12C | 0.7027 | 0.2479 | 0.0658 | 0.105* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C11 | 0.0725 (7) | 0.1143 (9) | 0.0598 (6) | -0.0115 (6) | 0.0303 (5) | 0.0005 (5) |
| O1 | 0.0435 (12) | 0.0732 (15) | 0.0515 (13) | -0.0175 (11) | 0.0154 (10) | -0.0114 (12) |
| N2 | 0.0440 (15) | 0.0478 (17) | 0.0599 (17) | -0.0044 (12) | 0.0146 (13) | -0.0034 (12) |
| N3 | 0.0401 (14) | 0.0422 (15) | 0.076 (2) | 0.0033 (12) | 0.0152 (13) | 0.0009 (13) |
| N1 | 0.0462 (15) | 0.0455 (16) | 0.0596 (18) | -0.0065 (13) | 0.0151 (13) | -0.0003 (13) |
| C7 | 0.0357 (15) | 0.054 (2) | 0.0471 (18) | -0.0056 (15) | 0.0090 (13) | -0.0038 (15) |
| C10 | 0.0344 (15) | 0.052 (2) | 0.062 (2) | -0.0015 (15) | 0.0148 (14) | -0.0025 (16) |
| O2 | 0.0556 (14) | 0.0429 (14) | 0.133 (2) | 0.0071 (11) | 0.0413 (15) | 0.0007 (15) |
| C6 | 0.0377 (16) | 0.0433 (18) | 0.0497 (19) | -0.0055 (13) | 0.0106 (14) | -0.0037 (14) |
| C1 | 0.0454 (18) | 0.0468 (18) | 0.056 (2) | -0.0005 (14) | 0.0155 (15) | 0.0048 (15) |
| C8 | 0.0326 (14) | 0.0462 (18) | 0.0449 (18) | -0.0034 (13) | 0.0070 (13) | -0.0017 (14) |
| C9 | 0.0363 (15) | 0.0460 (18) | 0.0424 (17) | -0.0036 (13) | 0.0072 (13) | -0.0028 (14) |
| C5 | 0.0478 (19) | 0.076 (2) | 0.056 (2) | -0.0112 (17) | 0.0200 (16) | -0.0001 (18) |
| C2 | 0.0400 (18) | 0.074 (2) | 0.074 (3) | -0.0041 (17) | 0.0190 (17) | 0.009 (2) |
| C4 | 0.057 (2) | 0.092 (3) | 0.050 (2) | -0.005 (2) | 0.0039 (17) | 0.0002 (19) |
| C11 | 0.0390 (16) | 0.063 (2) | 0.057 (2) | -0.0041 (15) | 0.0141 (14) | -0.0010 (17) |
| C3 | 0.0366 (18) | 0.081 (3) | 0.073 (3) | -0.0074 (17) | 0.0067 (17) | 0.002 (2) |
| C12 | 0.073 (2) | 0.052 (2) | 0.087 (3) | -0.0079 (18) | 0.026 (2) | 0.0095 (19) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-----------|-----------|----------|-----------|
| C11—C1 | 1.735 (3) | C8—C9 | 1.415 (4) |
| O1—C7 | 1.371 (3) | C9—C11 | 1.501 (4) |
| O1—C6 | 1.396 (3) | C5—C4 | 1.389 (5) |
| N2—C9 | 1.330 (4) | C5—H5 | 0.9300 |
| N2—N1 | 1.369 (3) | C2—C3 | 1.362 (5) |
| N3—C10 | 1.267 (4) | C2—H2A | 0.9300 |
| N3—O2 | 1.397 (3) | C4—C3 | 1.379 (5) |
| N1—C7 | 1.339 (4) | C4—H4 | 0.9300 |
| N1—C12 | 1.456 (4) | C11—H11A | 0.9600 |
| C7—C8 | 1.376 (4) | C11—H11B | 0.9600 |
| C10—C8 | 1.446 (4) | C11—H11C | 0.9600 |
| C10—H10 | 0.9300 | C3—H3 | 0.9300 |
| O2—H2 | 0.8200 | C12—H12A | 0.9600 |
| C6—C5 | 1.364 (4) | C12—H12B | 0.9600 |
| C6—C1 | 1.383 (4) | C12—H12C | 0.9600 |
| C1—C2 | 1.381 (5) | | |
| C7—O1—C6 | 117.8 (2) | C6—C5—C4 | 119.5 (3) |
| C9—N2—N1 | 106.1 (2) | C6—C5—H5 | 120.2 |
| C10—N3—O2 | 111.1 (3) | C4—C5—H5 | 120.2 |
| C7—N1—N2 | 109.8 (2) | C3—C2—C1 | 120.5 (3) |

| | | | |
|------------|-----------|---------------|-----------|
| C7—N1—C12 | 129.1 (3) | C3—C2—H2A | 119.8 |
| N2—N1—C12 | 121.1 (3) | C1—C2—H2A | 119.8 |
| N1—C7—O1 | 121.3 (3) | C3—C4—C5 | 120.4 (3) |
| N1—C7—C8 | 109.6 (3) | C3—C4—H4 | 119.8 |
| O1—C7—C8 | 128.9 (3) | C5—C4—H4 | 119.8 |
| N3—C10—C8 | 123.0 (3) | C9—C11—H11A | 109.5 |
| N3—C10—H10 | 118.5 | C9—C11—H11B | 109.5 |
| C8—C10—H10 | 118.5 | H11A—C11—H11B | 109.5 |
| N3—O2—H2 | 109.5 | C9—C11—H11C | 109.5 |
| C5—C6—C1 | 120.2 (3) | H11A—C11—H11C | 109.5 |
| C5—C6—O1 | 124.2 (3) | H11B—C11—H11C | 109.5 |
| C1—C6—O1 | 115.6 (3) | C2—C3—C4 | 119.7 (3) |
| C6—C1—C2 | 119.8 (3) | C2—C3—H3 | 120.2 |
| C6—C1—C11 | 119.7 (2) | C4—C3—H3 | 120.2 |
| C2—C1—C11 | 120.6 (3) | N1—C12—H12A | 109.5 |
| C7—C8—C9 | 103.5 (3) | N1—C12—H12B | 109.5 |
| C7—C8—C10 | 124.5 (3) | H12A—C12—H12B | 109.5 |
| C9—C8—C10 | 132.0 (3) | N1—C12—H12C | 109.5 |
| N2—C9—C8 | 111.0 (3) | H12A—C12—H12C | 109.5 |
| N2—C9—C11 | 120.3 (3) | H12B—C12—H12C | 109.5 |
| C8—C9—C11 | 128.6 (3) | | |

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> |
|-------------------------|-------------|---------------|-----------------------|-------------------------|
| O2—H2...N2 ⁱ | 0.82 | 1.97 | 2.787 (3) | 171 |

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