Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(2,4-Dichlorophenoxy)-*N*-(4,6-dimethylpyridin-2-yl)acetamide

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Key indicators

Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.109
Data-to-parameter ratio = 13.1

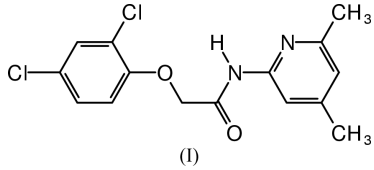
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 16 July 2003
Accepted 24 July 2003
Online 15 August 2003

The structure of the title compound, $C_{15}H_{14}Cl_2N_2O_2$, comprises an essentially flat molecule [dihedral angle of 3.64 (9)° between the two aromatic rings] with an intramolecular hydrogen-bonding interaction from the amine N—H group to the phenoxy O atom. Molecules are arranged in lamellar sheets parallel to the (110) plane.

Comment

A search of the April 2003 release of the Cambridge Structural Database (Allen, 2002) reveals that there are eight structures based on *N*-(4,6-dimethylpyridin-2-yl)carboxamide, all published by one research group. These structures are *N*-(4,6-dimethylpyridin-2-yl)-2-(3-nitrophenyl)acetamide (Rodier *et al.*, 1986), *N*-(4,6-dimethylpyridin-2-yl)-2-(4-nitrophenyl)propionamide (Rodier, Robert & Le Baut, 1990), (*E*)-*N*-(4,6-dimethylpyridin-2-yl)-3-phenylpropenamide hemihydrate (Rodier, Robert-Piessard & Le Baut, 1990), *N*-(4,6-dimethylpyridin-2-yl)-2-furamide (Rodier *et al.*, 1991), *N*-(4,6-dimethylpyridin-2-yl)-2-thiophenecarboxamide (Rodier, Cense *et al.*, 1992), *N*-(4,6-dimethylpyridin-2-yl)(1-methylindol-2-yl)-carboxamide (Rodier, Robert & Le Baut, 1992), *N*-(4,6-dimethylpyridin-2-yl)-2-(3-nitrophenyl)acetamide (Rodier *et al.*, 1993) and *N*-(4,6-dimethylpyridin-2-yl)-5-methylpyrazine-2-carboxamide (Rodier *et al.*, 1994). In a series of studies on the syntheses of additional derivatives of *N*-(4,6-dimethylpyridin-2-yl)carboxamide and also *N*-(4,6-dimethylpyrimidin-2-yl)-carboxamide as potential *anti*-inflammatory agents, we prepared the title compound, (I), and its structure is reported here.



(I)

The structure of (I) comprises an essentially flat molecule (Fig. 1) with an intramolecular hydrogen-bonding interaction from the amine N—H group to the phenoxy O atom (Table 1). Molecules of (I) are arranged in lamellar sheets parallel to the (110) plane. The dihedral angle between the substituted phenyl and pyridyl rings is 3.64 (9)°.

Experimental

Four molar equivalents of oxalyl chloride (2.29 g, 18.1 mmol) were added dropwise to a stirred solution of (2,4-dichlorophenoxy)acetic acid (1.0 g, 4.5 mmol) and a catalytic amount of dry pyridine (dried over KOH) in 20 ml dry dichloromethane (dried over molecular sieves). After stirring for 30 min, the solvent was removed under

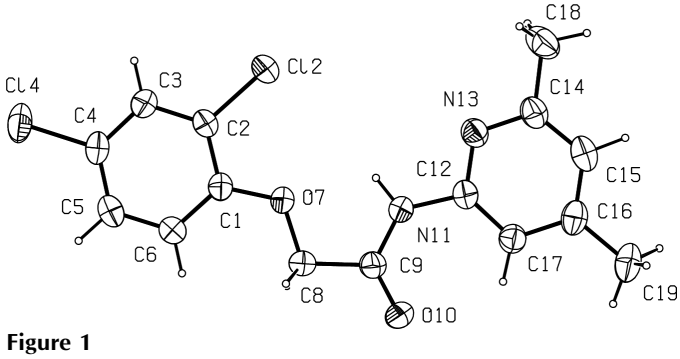


Figure 1
The molecular configuration and atom-numbering scheme for the title compound, showing 50% probability displacement ellipsoids.

reduced pressure, yielding a yellow solution of (2,4-dichlorophenoxy)acetic chloride. This solution was then added dropwise to a stirred solution of 2-amino-4,6-dimethylpyridine (0.55 g, 4.5 mmol) and triethylamine (0.46 g, 4.5 mmol) in 20 ml dry dichloromethane. After 30 min, the solution was filtered and the solvent removed under reduced pressure, yielding a white powder. The product was purified using column chromatography (SiO_2) and collected in the eluted ethyl acetate fraction after initial chloroform elutions. The solid product was further washed with 30 ml cold ethanol to afford 0.82 g of (I) (81%); m.p. 435–436 K; ^{1}H NMR ($CDCl_3$, 250 MHz): δ (p.p.m.) 2.33 (*s*, 3H, CH_3), 2.42 (*s*, 3H, CH_3), 4.62 (*s*, 2H, CH_2), 6.77 (*s*, 1H, Ar–H), 6.89 (*d*, J = 8.8 Hz, 1H, Ar–H), 7.33 (*d*, J = 8.8 Hz, 1H, Ar–H), 7.41 (*d*, J = 2.5 Hz, 1H, py–H), 7.87 (*s*, 1H, py–H), 8.89 (*bs*, 1H, NH); *m*/*z* 325 (MH^+, 100%), 327 (MH^+, 75%). Crystals suitable for X-ray diffraction studies were grown from ethyl acetate.

Crystal data

$C_{15}H_{14}Cl_2N_2O_2$
M_r = 325.18
Triclinic, $P\overline{1}$
a = 7.445 (5) Å
b = 8.790 (6) Å
c = 12.549 (7) Å
α = 89.28 (4)°
β = 87.08 (5)°
γ = 68.74 (4)°
V = 764.4 (9) Å^3
Z = 2
D_x = 1.413 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 reflections
θ = 15–16°
μ = 0.43 mm^{-1}
T = 220 (2) K
Prism, colourless
0.70 × 0.47 × 0.38 mm

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
3678 measured reflections
2570 independent reflections
2032 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.0°
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.05
2570 reflections
196 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.1634P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11···O7	0.87	2.07	2.552 (3)	114

All H atoms were included in the refinement at calculated positions, as riding atoms, with N—H set to 0.87 Å and C—H set to 0.97 (CH_3), 0.98 (CH_2) or 0.94 Å (Ar–H); the isotropic displacement parameters were set at 1.25 times U_{eq} of the carrier atom.

Data collection: *DIF*4 (Stoe & Cie, 1990); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

The authors thank the EPSRC Chemical Database Service at Daresbury for access to the Cambridge Structural Database.

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