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2-(2,4-Dichlorophenoxy)-*N*-(4,6-dimethoxypyrimidin-2-yl)propionamide

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.057
wR factor = 0.173
Data-to-parameter ratio = 13.6

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

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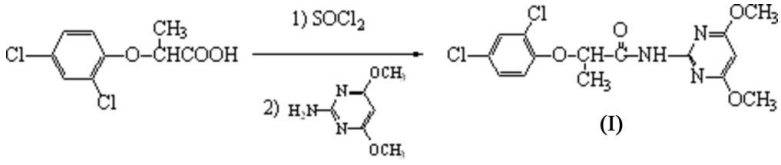
The title compound, $C_{15}H_{15}Cl_2N_3O_4$, is an amide herbicide with a pyrimidine ring attached to the N atom of the CONH group. The crystal structure determination reveals that there is an intramolecular hydrogen bond, forming a five-membered ring.

Comment

Some amide compounds show high biological herbicidal activities with low toxicity (Katsushi *et al.*, 1996; Kenji *et al.*, 1992; Nobuyuki *et al.*, 1990; Michaely & Knudsen, 1986). Investigations using amides as herbicides have become the subject of intensive research and many novel structural phenoxyalkanoic acid amide herbicides have appeared in the literature (Whang *et al.*, 2002, 2003; Yuji *et al.*, 2000; Keiji *et al.*, 1990; Foerster *et al.*, 1985). Although some of the phenoxyalkanoic acid amides have been described, so far, relatively few reports on the crystal structures of amides with a pyrimidine ring are available (Tetsuo *et al.*, 1987). We report here the synthesis and crystal structure of 2-(2,4-dichlorophenoxy)-*N*-(4,6-dimethoxypyrimidin-2-yl)propionamide, (I). The key feature of this amide herbicide is that the 2,4-dichlorophenoxypropionyl group is connected to a pyrimidine ring, substituted in both *meta* positions, by an amide link, which might provide an opportunity for the study of cooperative effects between the two types of biologically active groups.



The molecular structure of the title compound is shown in Fig. 1. An intramolecular hydrogen bond (N1—H1···O1) forms a five membered ring (Table 2).

Experimental

Under a nitrogen atmosphere, a stirred solution of 2,4-dichlorophenoxypropionic acid (0.50 g, 2.14 mmol) in $SOCl_2$ (5 ml) was refluxed for 4 h. After removing the $SOCl_2$, to the resulting acid chloride was added a solution of 4,6-dimethoxy-2-aminopyrimidine (0.33 g, 2.14 mmol) and triethylamine (0.22 g, 2.14 mmol) in dry CH_2Cl_2 (5 ml) at 273 K over 20 min. The mixture was refluxed and stirred for another 2 h. After evaporation of most of the solvent, the residue was cooled to room temperature and water (5 ml) was added to quench the reaction. The residue was repeatedly extracted with 50 ml of diethyl ether. The combined organic layer was washed with water and brine, and then dried over Na_2SO_4. After evaporation of

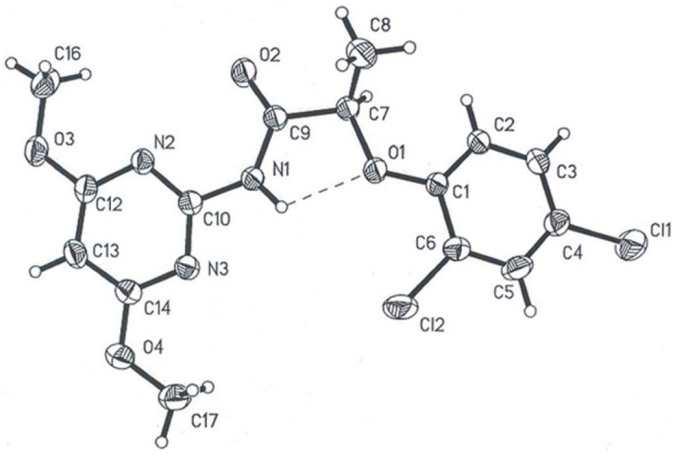


Figure 1
The structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed line represents the hydrogen bond.

solvent, the residue was further purified by column chromatography on silica gel (hexane–ethyl acetate 2:1) to give a white crystalline solid in 65% isolated yield. Crystals suitable for single-crystal X-ray diffraction were obtained by cooling the hot mixture solution of ethyl acetate–hexane (1:1 *v*/*v*) (yield: 65%; m.p. 371–373 K). ^{1}H NMR (DMSO-d_6, 400 MHz): δ 10.71 (*s*, 1H, NH), 7.61–6.96 (*m*, 3H, Ph—H), 5.97 (*s*, 1H, py—H), 5.39–5.38 (*q*, 1H, $CHCH_3$), 3.87 (*s*, 6H, OCH_3), 1.59–1.58 (*d*, 3H, $CHCH_3$); IR (KBr): 811, 1196, 1719, 3391 cm^{-1}; MS(EI) (70 eV) *m*/*z* (%): 373 (3.29) $[M+2]^+$, 371 (3.11) $[M]^+$, 210 (100), 155 (39.33); Analysis calculated for $C_{15}H_{15}Cl_2N_3O_4$: C 48.40, H 4.06, N 11.29%; found: C 48.51, H 4.08, N 11.28%.

Crystal data

$C_{15}H_{15}Cl_2N_3O_4$
M_r = 372.20
Triclinic, $P\overline{1}$
a = 8.345 (2) Å
b = 9.944 (4) Å
c = 11.779 (3) Å
α = 84.580 (7)°
β = 71.698 (5)°
γ = 70.013 (4)°
V = 872.0 (5) Å^3
Z = 2
D_x = 1.421 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3006 reflections
θ = 2.0–25.0°
μ = 0.40 mm^{-1}
T = 298 (2) K
Block, colorless
0.35 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.874, T_{max} = 0.962
3645 measured reflections
3006 independent reflections
2150 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.0°
$h = -7 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.173
S = 0.98
3006 reflections
221 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0917P)^2 + 0.5362P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.009 (1)

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.740 (3)	O1—C7	1.439 (3)
Cl2—C6	1.735 (3)	O2—C9	1.206 (4)
N1—C9	1.353 (4)	O3—C16	1.425 (5)
N1—C10	1.395 (4)	O4—C17	1.432 (4)
C9—N1—C10	129.8 (3)	O2—C9—N1	126.0 (3)
C1—O1—C7	119.2 (2)	O2—C9—C7	118.6 (3)
O1—C7—C9	107.8 (2)	N1—C9—C7	115.4 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	2.07	2.551 (3)	115

The H atoms were positioned geometrically (C—H = 0.93, 0.96 or 0.97 Å and N—H = 0.86 Å) and refined using the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (1998). *SMART*, *SAINT*, *SHELXTL* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Foerster, H. & Eue, L. (1985). US Patent No. A01N37/20, 4500341, 1985–02–19.
Katsushi, M. & Masatoshi, O. (1996). US Patent No. A01N43/56, 5545608, 1996–08–13.
Keiji, T. & Kuniaki, S. (1990). JP Patent No. JP2011580, 1990-01-16.
Kenji, H. & Kazumi, N. (1992). JP Patent No. A01N37/18, 4210949, 1992–08–03.
Michaely, W. & Knudsen, C. (1986). US Patent No. A01N37/34, 4623384, 1986–11–18.
Nobuyuki, O. & Atsuhiko, I. (1990). EP Patent No. A01N43/36, 0372586, 1990–06–13.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Göttingen, Germany.
Tetsuo, T. & Yasutomo, T. (1987). JP Patent No. A01N43/54, 62042974, 1987–02–24.
Whang, K. D. & Sung, C. H. (2002). US Patent No. A01N43/76, 6486098, 2002–11–26.
Whang, K. D. & Sung, C. H. (2003). US Patent No. A01N43/76, 2003096706, 2003–05–22.
Yuji, A. & Isamu, N. (2000). JP Patent No. JP2000086602, 2000-03-28.