

Methyl 4-{2-chloro-4-[2-(4,6-dimethoxy-
pyrimidin-2-yloxy)benzylamino]phenoxy}-
benzoateZheng-Bo Chen,^a Jun Wu,^{a*}
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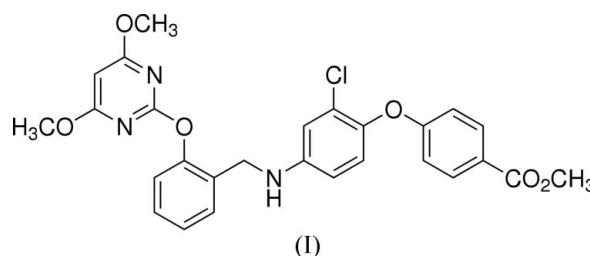
Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.046
 wR factor = 0.136
Data-to-parameter ratio = 16.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, $\text{C}_{27}\text{H}_{24}\text{ClN}_3\text{O}_6$, a probable new herbicide, was synthesized by the reaction of 4,6-dimethoxy-2-(methylsulfonyl)pyrimidine and methyl 4-[2-chloro-4-(2-hydroxybenzylamino)phenoxy]benzoate in tetrahydrofuran. π - π Interactions occur between neighbouring benzene rings.

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Comment

4,6-Dimethoxy-2-phenoxy pyrimidine and its derivatives have shown remarkable herbicidal activities in recent years (Nezu *et al.*, 1996; Tamaru *et al.*, 1997; Hudson *et al.*, 2002). The title compound, (I), has been prepared as a new herbicide.The partially overlapped arrangement between adjacent parallel C3-benzene rings and the face-to-face distance of 3.63 (1) Å indicate the existence of π - π aromatic stacking interactions (Fig. 2). Neighbouring molecules are also linked to each other *via* N—H \cdots O hydrogen bonds (Table 1 and Fig. 3).

Experimental

A mixture of methyl 4-[2-chloro-4-(2-hydroxybenzylamino)phenoxy]benzoate (0.75 g, 0.002 mol), 4,6-dimethoxy-2-(methylsulfonyl)pyrimidine (0.43 g, 0.002 mol), and K_2CO_3 (0.55 g, 0.004 mol) in tetrahydrofuran (50 ml) was refluxed for 8 h. After the insoluble substance was removed from the mixture by filtration, the organic layer was evaporated *in vacuo* to give the crude product. Recrystallization of the crude product from ethanol gave colorless crystals (m.p. 387.2–388.0 K).

Crystal data

 $\text{C}_{27}\text{H}_{24}\text{ClN}_3\text{O}_6$
 $M_r = 521.94$
Triclinic, $P\bar{1}$
 $a = 10.763$ (4) Å
 $b = 11.066$ (6) Å
 $c = 12.051$ (4) Å
 $\alpha = 98.02$ (3)°
 $\beta = 116.33$ (3)°
 $\gamma = 94.746$ (19)° $V = 1256.8$ (10) Å³
 $Z = 2$
 $D_x = 1.379$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.20$ mm⁻¹
 $T = 298$ (1) K
Block, colorless
0.35 × 0.28 × 0.18 mm

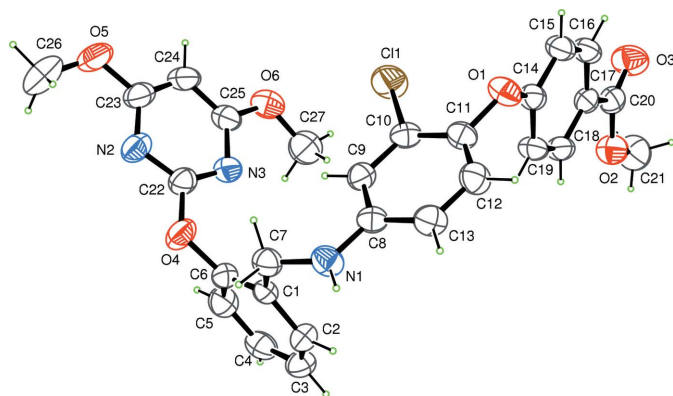


Figure 1
The molecular structure of (I) with 40% probability displacement ellipsoids. H atoms are shown as circles of arbitrary radii.

Data collection

Rigaku R-AXIS RAPID diffractometer
 ω scans
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.915$, $T_{\max} = 0.965$

12447 measured reflections
 5677 independent reflections
 3660 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.136$
 $S = 1.08$
 5677 reflections
 337 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0686P)^2 + 0.0343P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.44 \text{ e } \text{\AA}^{-3}$

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O3^i$	0.92	2.30	3.126 (3)	150
$C7-H7A\cdots O4$	0.97	2.49	2.828 (3)	100

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

Atom H1 was located in a difference Fourier map and refined as riding in its as-found relative position, with a fixed $U_{\text{iso}}(\text{H})$ value of 0.08 \AA^2 . Other H atoms were placed in calculated positions, with $C-H = 0.93$ (aromatic), 0.96 (methyl group) and 0.97 \AA (methylene group), and were refined in riding mode, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ (aromatic and methylene) and $1.5U_{\text{eq}}$ (methyl).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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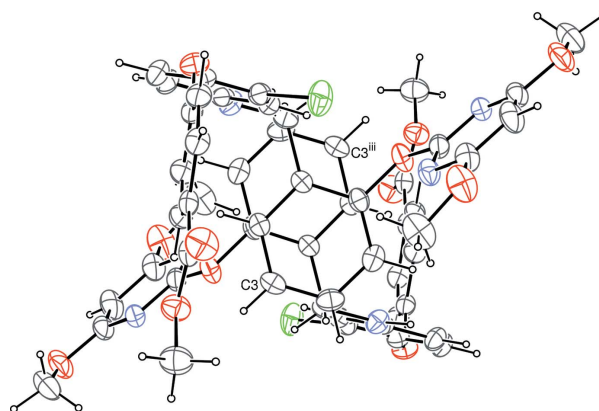


Figure 2
 π - π Stacking between parallel C1-C6 and C1-C6ⁱⁱⁱ rings of neighbouring molecules; atoms C3 and C3ⁱⁱⁱ are labelled [Symmetry code: (iii) $2 - x, 2 - y, -z$].

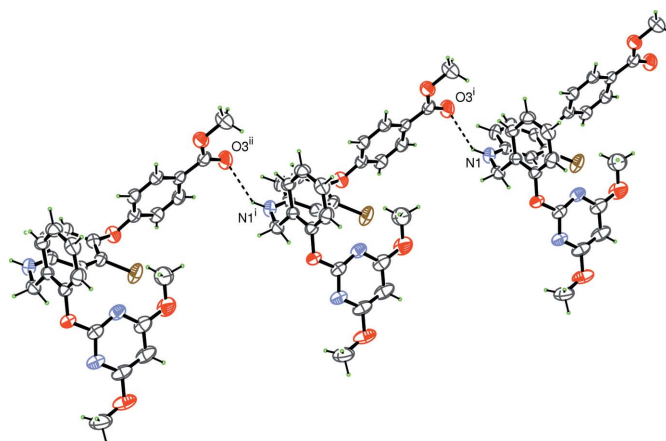


Figure 3
The intermolecular hydrogen bonding (dashed lines) in the crystal structure. [Symmetry codes: (i) $x, y, z - 1$; (ii) $x, y, z - 2$.]

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