Methyl 2,6-bis(4,6-dimethoxypyrimidin-2-yloxy)benzoate

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In the title compound, $C_{20}H_{20}N_4O_8$, there are intermolecular C—H···O hydrogen bonds and π–π stacking interactions.

Comment

KIH-2023, a phenoxypyrimidine derivative, is a highly effective herbicide (Wada *et al.*, 1988, 1990, 1992). In this paper, we report the result of an X-ray diffraction study of the title compound, (I), which has a similar structure to the herbicide KIH-2023.

(I)
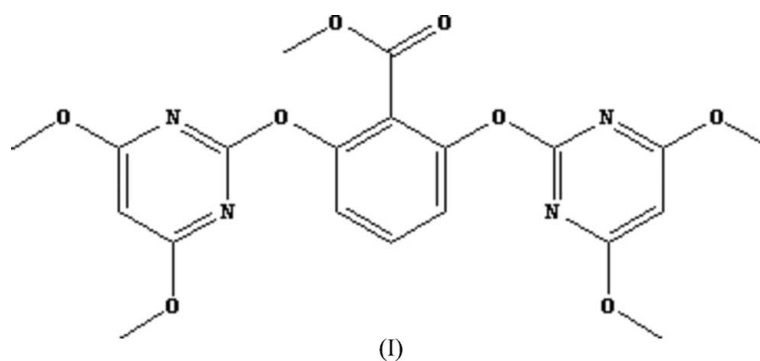
The two planar pyrimidine rings are twisted (Fig. 1), the angle between them being 87.6 (2)°. The dihedral angles between the benzene ring and the pyrimidine (N1/C3–C5/N2/C6) and (N3/C15/N4/C16–C18) rings are 65.7 (2) and 58.1 (2)°, respectively. The crystal packing is reinforced by an intermolecular C—H···O hydrogen bond (Table 2 and Fig. 2) and a π–π stacking interaction between the benzene rings [C7–C12 and C7^{ii}–C12^{ii}; symmetry code: (ii) $-x$, $2 - y$, $-z$] (Fig. 3). The centroid–centroid distance is 3.917 (4) Å and the interplanar spacing is 3.398 (4) Å.

Experimental

2,6-Dihydroxybenzoic acid (15.4 g, 0.1 mol) in methanol (60 ml) with H_2SO_4 (3.5 ml) was refluxed for 34 h. The excess methanol was removed under reduced pressure and 300 ml water was then added. The precipitate was filtered off and dried at room temperature. The product was recrystallized from cyclohexane to give methyl 2,6-dihydroxybenzoate (7.3 g, yield 43.5%). To a solution of this ester (3 g, 17.8 mmol) in acetone (50 ml) was added K_2CO_3 (6.16 g, 44.6 mmol) and the mixture was refluxed for 1 h. 2-Methanesulfonyl-4,6-dimethoxypyrimidine (7.79 g, 35.73 mmol) was added and the mixture was refluxed for 5 h. The precipitate was filtered off and dried under reduced pressure to give a white solid which was purified by recrystallization from ethanol (Yield 7.25 g, 91.5%). Crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution at 298 K.
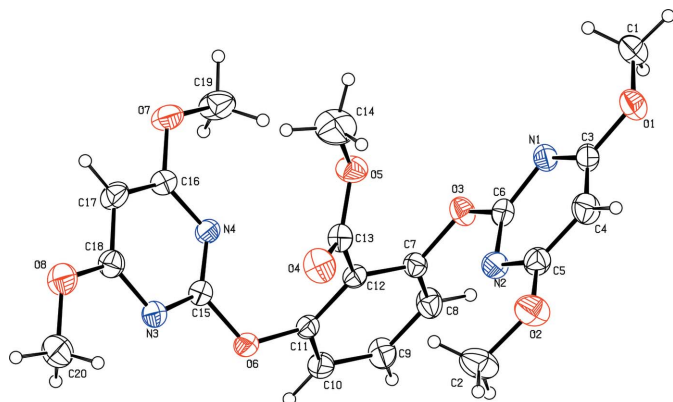
Figure 1
A view of (I), showing the atom-labeling scheme. Displacement ellipsoids
are drawn at the 50% probability level.

Crystal data

$C_{20}H_{20}N_4O_8$
$M_r = 444.40$
Monoclinic, $C2/c$
$a = 29.00\ (3)$ Å
$b = 7.776\ (7)$ Å
$c = 22.34\ (2)$ Å
$\beta = 122.753\ (16)°$
$V = 4237\ (7)$ Å^3
$Z = 8$

$D_x = 1.393$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1421
 reflections
$\theta = 2.8–21.6°$
$\mu = 0.11$ mm^{-1}
$T = 292\ (2)$ K
Block, colorless
$0.30 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART 4K CCD area-
 detector diffractometer
φ and ω scans
Absorption correction: none
11217 measured reflections
4161 independent reflections

2544 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 26.0°$
$h = -21 \rightarrow 35$
$k = -9 \rightarrow 9$
$l = -27 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.129$
$S = 0.99$
4161 reflections
294 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.440 (3)	C6—O3	1.364 (3)
C3—O1	1.344 (3)	C13—O4	1.194 (3)
C6—N1	1.325 (3)	C13—O5	1.337 (3)
N2—C6—O3	118.5 (2)	C12—C7—O3	122.2 (2)
N1—C6—O3	111.9 (2)	O5—C13—C12	111.4 (2)
C8—C7—O3	115.8 (2)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C14—H14C$\cdots$O7^i	0.96	2.60	3.444 (4)	147

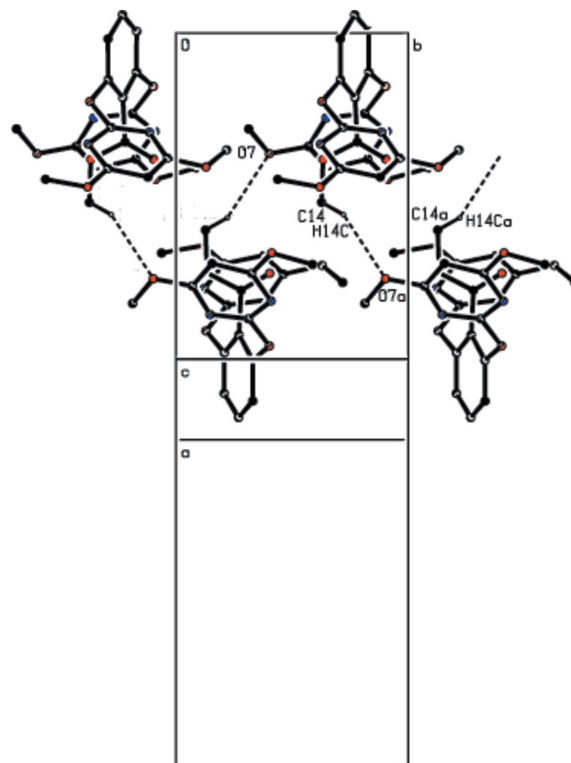Symmetry code: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

Figure 2
C—H$\cdots$O hydrogen bonds in (I). Hydrogen bonds are shown as dashed
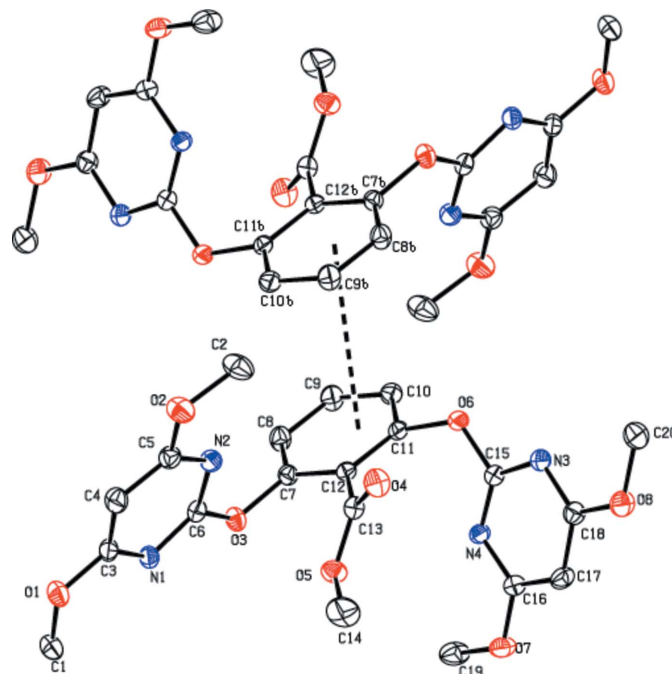lines. The suffix a corresponds to symmetry code (i) in Table 2.

Figure 3
A π–π stacking interaction (dashed line) in (I). The suffix b corresponds
to symmetry code $(-x, 2 - y, -z)$.

H atoms were placed at calculated positions and refined as riding
(C—H = 0.93–0.96 Å), with U_{iso}(H) = $1.2U_{eq}$(C) or $1.5U_{eq}$(methyl C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); 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, 2001); software used to prepare material for publication: *SHELXTL*.

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