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5-(4-Methoxybenzoyl)-4-(4-methoxyphenyl)-1-[1-(4-methoxyphenyl)ethylidenenamino]-pyrimidin-2(1*H*)-one

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.143
Data-to-parameter ratio = 13.0

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

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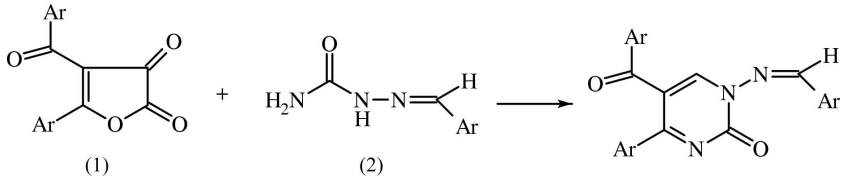
In the title compound, $C_{27}H_{23}N_3O_5$, the pyrimidine ring is slightly puckered. The mean plane of the pyrimidine ring forms dihedral angles of 28.26 (10), 55.53 (9) and 55.96 (10)° with the three benzene rings in the molecule. There are intramolecular and intermolecular C—H···O interactions.

Comment

Pyrimidines in general have attracted much interest for biological and medicinal reasons. In particular, various analogues of pyrimidines possess effective antibacterial, antifungal, antiviral, insecticidal and miticidal activities (Cheng, 1969; McNair-Scott *et al.*, 1959; Sankyo Co. Ltd/Ube Industries Ltd, 1984; Kollenz, 1972; Maslivets *et al.*, 1988; Kruglenko *et al.*, 1987; Kozlov *et al.*, 1986). Conformational analysis and quantum chemical calculations have also been carried out by means of semi-empirical calculations for a series of functionalized 1*H*-pyrimidines (Yıldırım *et al.*, 1995, 1996; Sarıpınar *et al.*, 1996). In the present study, we have carried out the reaction of the furan-2,3-dione (1), which is obtained from *p,p′*-dimethoxydibenzoylmethane and oxalyl dichloride (Sarıpınar *et al.*, 2000), with the semicarbazone (2) to obtain the 1,4,5-trisubstituted 1*H*-pyrimidine-2-one (3) (see scheme).



(1) (2) (3)

Ar = p−$CH_3OC_6H_4$−

Fig. 1 shows the molecular structure and conformation of (3) with the atomic numbering scheme. Although close to being planar, the pyrimidine ring is slightly puckered. The angle between the planes formed by the ring atoms N2—C3—C4 and C2—N1—C1 is 7.7 (4)°. The corresponding angle in 1-allyl-5-benzoyl-4-phenylpyrimidin-2-one (Öztürk *et al.*, 1997) is 9.0 (7)°. Selected bond lengths, angles and torsion angles are presented in Table 1. In the compound, the *A* (C13–C18), *B* (C6–C11) and *C* (C21–C26) benzene rings form dihedral angles of 28.26 (10), 55.53 (9) and 55.96 (10)° with the pyrimidine ring, respectively. There are weak C—H···O interactions (Table 2), but no π–π or C—H···π interactions.

Experimental

For the preparation of the title compound, 4-(4-methoxybenzoyl)-5-(4-methoxyphenyl)furan-2,3-dione, (1) (0.32 g, 0.95 mmol), and *p*-methoxybenzaldehyde semicarbazone, (2) (0.18 g, 0.95 mmol), were

heated at 413 K for 1 h without any solvent. After cooling to room temperature, the residue was treated with dry diethyl ether and the crude product formed was crystallized from ethanol and dried over P_2O_5 (yield: 0.2 g, 45%; m.p. 490 K). IR (KBr, ν, cm^{-1}): 3450 (C═O, carbonyl overtone), 3050 (aromatic C—H), 1665 and 1650 (C═O). ^{1}H NMR ($CDCl_3$): δ 9.68 (*s*, 1H, —N═C—H), 8.30 (*s*, 1H, C6—H), 6.87–6.74 (*m*, 12H, ArH), 3.82, 3.76 and 3.68 (*q*, 9H, —OCH_3). ^{13}C NMR ($CDCl_3$): δ 192.44 (*t*, ArCO), 171.56 (*s*, C-4), 153.57 (*s*, C-2), 150.72 (*s*, C-6), 135.02 (*s*, C-8), 134.45–115.78 (*m*, aromatic C), 118.68 (*s*, C-5), 57.58, 57.46 and 57.40 (*q*, 3CH_3O). Analysis calculated for $C_{27}H_{23}N_3O_5$: C 69.06, H 4.94, N 8.95%; found: C 69.20, H 5.19, N 8.88%.

Crystal data

$C_{27}H_{23}N_3O_5$
M_r = 469.48
Triclinic, $P\overline{1}$
a = 8.1127 (7) Å
b = 10.9405 (9) Å
c = 13.9263 (11) Å
α = 77.147 (7)°
β = 83.542 (7)°
γ = 76.749 (7)°
V = 1170.54 (17) Å^3
Z = 2
D_x = 1.332 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15633 reflections
θ = 2.0–28.8°
μ = 0.09 mm^{-1}
T = 296 K
Plate, colorless
0.46 × 0.27 × 0.07 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.965, T_{max} = 0.994
12784 measured reflections
4119 independent reflections
2655 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 25.0°
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.143
S = 1.01
4119 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0787P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.215 (3)	N3—C20	1.278 (3)
N1—C1	1.316 (3)	C1—C4	1.430 (3)
N1—C2	1.362 (3)	C3—C4	1.359 (3)
N2—C3	1.337 (3)	C4—C5	1.498 (3)
N2—C2	1.419 (3)		
C1—C4—C5	127.80 (19)	N3—C20—C21	121.0 (2)
O3—C9—C10	125.7 (3)	C22—C21—C20	122.6 (2)
C14—C13—C1	122.69 (18)		
C3—N2—N3—C20	−135.3 (2)	C4—C5—C6—C7	154.0 (2)
C3—C4—C5—C6	137.2 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C20—H20···O1	0.93	2.34	2.737 (3)	105
C11—H11···O4^{i}	0.93	2.60	3.512 (3)	166
C3—H3···O2^{ii}	0.93	2.54	3.330 (3)	143

Symmetry codes: (i) $x-1, y, z$; (ii) $-1-x, 1-y, 1-z$.

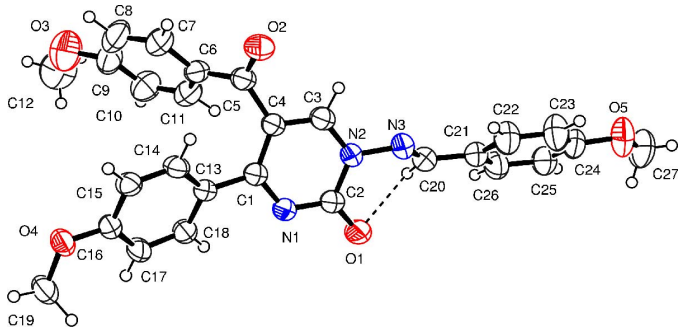


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of the structure of (3), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a C—H···O interaction.

H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.96 Å and with U_{iso}(H) = 1.2U_{eq}(C) (1.5U_{eq} for methyl).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); 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* publication routines (Farrugia, 1999).

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