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## Key indicators

Single-crystal X-ray study  
 $T = 296$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
Disorder in main residue  
 $R$  factor = 0.041  
 $wR$  factor = 0.089  
Data-to-parameter ratio = 12.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## 5-(4-Methoxybenzoyl)-4-(4-methoxyphenyl)-1-[1-(4-methoxyphenyl)ethylideneamino]-pyrimidin-2(1H)-one

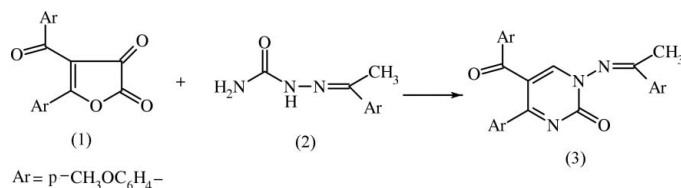
In the title compound,  $\text{C}_{28}\text{H}_{25}\text{N}_3\text{O}_5$ , the three benzene rings form dihedral angles of 58.09 (6), 36.97 (6) and 48.01 (7)° with the pyrimidine ring. One of the *p*-methoxy groups shows conformational disorder. A three-dimensional structure is formed *via* weak  $\text{C}-\text{H}\cdots\text{O}$  hydrogen-bond interactions.

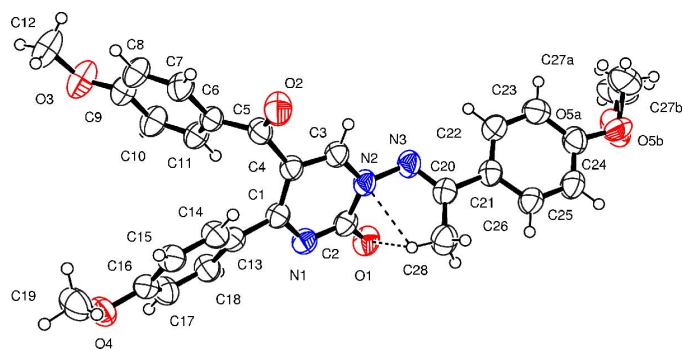
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## Comment

In relation to attempts to gain some insight into the chemical behaviour of five-membered heterocyclic 2,3-diones with NH-nucleophiles (Terpetschnig *et al.*, 1988; Akçamur *et al.*, 1986; Kollenz *et al.*, 1976; Ott *et al.*, 1976; Kollenz, 1972; Maslivets *et al.*, 1988; Kruglenko *et al.*, 1987; Kozlov *et al.*, 1986; Andreichikov *et al.*, 1987), a convenient preparation of functionalized 1*H*-pyrimidine-2-thiones from 4-benzoyl-5-phenyl-furan-2,3-dione and several thiosemicarbazones has been reported recently (Akçamur *et al.*, 1988; Altural *et al.*, 1989; Altural & Kollenz, 1990; Akkurt *et al.*, 1992, 2003). Pyrimidines in general are of great interest for biological and medicinal reasons, and thus the chemistry of these compounds has been investigated extensively. Various analogues of pyrimidines possess effective antibacterial, antifungal, antiviral, insecticidal and mitocidal activities (Cheng, 1969; McNair-Scott *et al.*, 1959; Sankyo Co. & Ube Industries, 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 semiempirical calculations for a series of functionalized 1*H*-pyrimidines (Yıldırım *et al.*, 1995; Sarıpınar *et al.*, 1996; Yıldırım *et al.*, 1996).In the present study, we carried out the reaction of the furan-2,3-dione, (1), which is obtained from *p,p'*-dimethoxy dibenzoylmethane and oxalyl dichloride (Sarıpınar *et al.*, 2000), with the semicarbazone, (2), and obtained the title 1,4,5-substituted 1*H*-pyrimidine-2-one, (3), in moderate yield (35%).In the structure of (3) (Fig. 1), the pyrimidine ring is slightly puckered, with puckering parameters (Cremer & Pople, 1975)  $Q_2 = 0.513$  (8) Å,  $Q_3 = 0.147$  (5) Å,  $Q_T = 0.533$  (9) Å,  $\theta = 73.97$  (4)° and  $\varphi_2 = 62.80$  (6)°. The benzene rings *A* (C13–C18), *B* (C6–C11) and *C* (C21–C26) form dihedral angles of

**Figure 1**

An ORTEP-3 (Farrugia, 1997) drawing of the molecular structure of (3), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The weak intramolecular C—H...O and C—H...N interactions are represented by dashed lines.

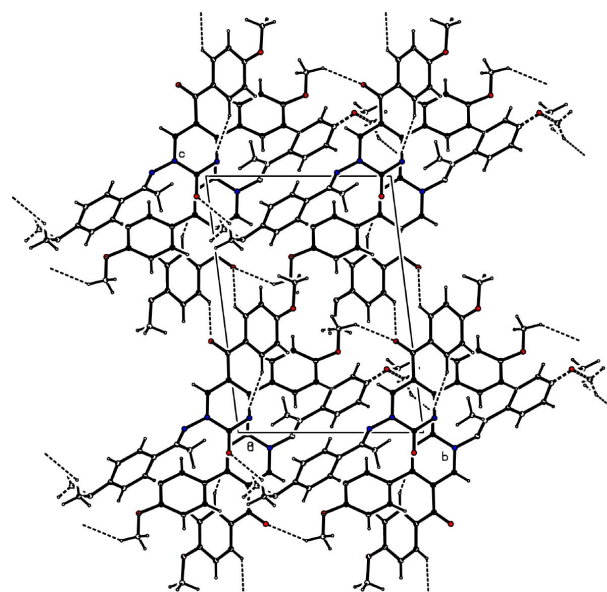
58.09 (6), 36.97 (6) and 48.01 (7)°, respectively, with the pyrimidine ring. The average C—N bond length in the pyrimidine ring is 1.375 (2) Å, compared with 1.403 (4) Å in 7-acetyl-5-benzoyl-6-phenyl-8-methyl-4,7-dihydropyrazolo[1,5-*c*]-1*H*-pyrimidine-2-one (Çelik *et al.*, 2000). Selected bond lengths of (3) are presented in Table 1. The weak intra- and intermolecular interactions in the structure are listed in Table 2. A three-dimensional structure is formed *via* weak intermolecular C—H...O interactions. There are no significant  $\pi$ - $\pi$  or C—H... $\pi$  interactions.

## Experimental

4-(4-Methoxybenzoyl)-5-(4-methoxyphenyl)furan-2,3-dione, (1) (0.28 g, 0.83 mmol), and *p*-methoxyacetophenonsemicarbazone, (2) (0.17 g, 0.83 mmol), were refluxed in toluene for 5 h. The solvent was evaporated and the remaining oily residue was treated with dry diethyl ether to give a white crude product, which was recrystallized from ethanol and allowed to dry over P<sub>2</sub>O<sub>5</sub> (yield 0.14 g, 35%; m.p. 479 K). IR: 3020 (aromatic C—H), 1660 and 1640 (C=O); <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  8.20 (s, 1H, C6—H), 8.05–6.60 (*m*, 12H, ArH), 3.85, 3.63 and 3.53 (9H, 3CH<sub>3</sub>O—), 2.43 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (DMSO):  $\delta$  191.86 (*t*, ArCO), 176.25 (*s*, C4), 153.29 (*s*, C2), 148.60 (*s*, C6), 135.15 (*s*, C8), 137.72–115.74 (*m*, aromatic C), 118.38 (*s*, C5), 57.55, 57.42 and 57.38 (*q*, 3CH<sub>3</sub>O), 19.71 (CH<sub>3</sub>). Analysis calculated for C<sub>28</sub>H<sub>25</sub>N<sub>3</sub>O<sub>5</sub> (483): C 69.54, H 5.21, N 8.70%; found: C 69.86, H 5.50, N 8.76%.

### Crystal data

C <sub>28</sub> H <sub>25</sub> N <sub>3</sub> O <sub>5</sub>	<i>Z</i> = 2
<i>M<sub>r</sub></i> = 483.51	<i>D<sub>x</sub></i> = 1.279 Mg m <sup>-3</sup>
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> $\alpha$ radiation
<i>a</i> = 8.9010 (10) Å	Cell parameters from 8135 reflections
<i>b</i> = 10.5060 (12) Å	$\theta$ = 2.0–28.8°
<i>c</i> = 14.5942 (15) Å	$\mu$ = 0.09 mm <sup>-1</sup>
$\alpha$ = 92.522 (9)°	<i>T</i> = 296 K
$\beta$ = 104.923 (8)°	Prism, colourless
$\gamma$ = 106.330 (8)°	0.29 × 0.20 × 0.07 mm
<i>V</i> = 1255.5 (3) Å <sup>3</sup>	

**Figure 2**

PLATON plot (Spek, 2003), illustrating the hydrogen-bonding network and the packing of molecules of (3). Hydrogen bonds are shown as dashed lines.

### Data collection

Stoe IPDS-II diffractometer	2541 reflections with <i>I</i> > 2σ( <i>I</i> )
$\omega$ scans	<i>R</i> <sub>int</sub> = 0.040
Absorption correction: integration ( <i>X-RED32</i> ; Stoe & Cie, 2002)	$\theta_{\max}$ = 25.0°
<i>T</i> <sub>min</sub> = 0.976, <i>T</i> <sub>max</sub> = 0.995	<i>h</i> = -9 → 10
11 921 measured reflections	<i>k</i> = -12 → 12
4430 independent reflections	<i>l</i> = -17 → 17

### Refinement

Refinement on <i>F</i> <sup>2</sup>	$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.089$	( $\Delta/\sigma$ ) <sub>max</sub> = 0.001
<i>S</i> = 1.00	$\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$
4430 reflections	$\Delta\rho_{\min} = -0.12 \text{ e \AA}^{-3}$
345 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.0085 (10)

**Table 1**

Selected interatomic distances (Å).

N1—C2	1.369 (2)	N2—C2	1.415 (2)
N2—C3	1.341 (2)		

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C28—H28A...O1	0.96	2.56	2.939 (3)	104
C28—H28A...N2	0.96	2.33	2.763 (3)	107
C27a—H27B...O1 <sup>i</sup>	0.96	2.52	3.308 (16)	140
C7—H7...O2 <sup>ii</sup>	0.93	2.58	3.358 (2)	142
C11—H11...O1 <sup>iii</sup>	0.93	2.47	3.307 (2)	150
C19—H19A...O2 <sup>iv</sup>	0.96	2.40	3.217 (3)	143

Symmetry codes: (i) *x*, *y* - 1, *z*; (ii) -*x* + 1, -*y* + 2, -*z* + 1; (iii) -*x* + 1, -*y* + 2, -*z*; (iv) *x*, *y* + 1, *z*.

Atoms O5 and C27 of the methoxy group are conformationally disordered over two site. The occupancies of the disordered positions

O5a, O5b, C27a and C27b were initially refined, and later fixed at 50% for O5a/C27a and 50% for O5b/C27b. H atoms were positioned geometrically and treated as riding, with C—H bond lengths of 0.93–0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  [ $1.5U_{\text{eq}}(\text{C})$  for methyl H atoms].

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) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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