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4,7-Bis(4-methoxyphenyl)-1,3,7-triphenyl-2,3,5,6,7,7a-hexahydro-1*H*-pyrrolo[2,3-*d*]-pyrimidine-2,5,6-trione

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.139
Data-to-parameter ratio = 16.3

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

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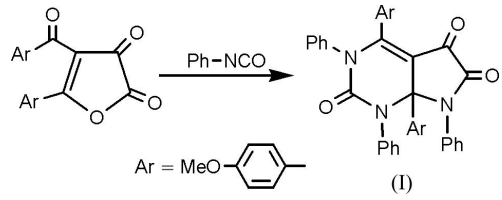
The synthesis of the title compound, $C_{38}H_{29}N_3O_5$, proceeds through a [4 + 2]-cycloaddition reaction. 4-(4-Methoxybenzoyl)-5-(4-methoxyphenyl)furan-2,3-dione was reacted with phenyl isocyanate to synthesize this new derivative of pyrrolo[2,3-*d*]pyrimidine in a low-temperature reaction. The molecule is composed of a pyrrolopyrimidine moiety with three phenyl and two *p*-methoxyphenyl substituents.

Comment

Some pyrrolo[2,3-*d*]pyrimidines are known to possess considerable antitumor, antiallergic, antiviral and anti-inflammatory activities (Hutzenlaub *et al.*, 1972; Smith *et al.*, 1972). As part of our interest in such compounds, we have synthesized and studied the single-crystal X-ray structure of the title compound, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. Its structure is similar to that of 7,7a-dihydro-1,3-bis(4-methylphenyl)-4,7,7a-triphenyl-1*H*-pyrrolo[2,3-*d*]pyrimidin-2,5,6-(3*H*)-trione, (II) (Kollenz *et al.*, 1984). However, the substitution on the pyrrolopyrimidine moiety differs. In (I), the substituents on atoms N1 and N2 are phenyl, whereas in (II) they are *p*-tolyl. The substituents on atoms C1 and C6 in (I) are *p*-methoxyphenyl, whereas in (II) these substituents are phenyl. The bond lengths and angles for the pyrrolopyrimidine skeleton of (I) and (II) are comparable.

In the crystal structure of (I), there are intermolecular C—H···O hydrogen-bonding interactions (Table 1).

Experimental

A mixture of 4-(4-methoxybenzoyl)-5-(4-methoxyphenyl)furan-2,3-dione (1 g, 2.96 mmol) and phenyl isocyanate (1.05 g, 8.88 mmol) was heated at 333–338 K for 24 h without any solvent in a 25 ml round-bottomed flask equipped with a calcium chloride guard tube. After cooling to room temperature, the residue was treated with anhydrous diethyl ether and the crude product recrystallized from acetic acid and ethanol to give yellow crystals of (I) (yield: 1.26 g, 70%; m.p. 474 K). IR (KBr): ν 1730, 1686, 674 cm^{-1} (C═O), 1579 cm^{-1} (C═C). ^1H NMR ($CDCl_3$): δ 7.67–6.20 (*m*, 23H, Ph), 3.89, 3.76 (*s*, 6H, CH_3O); ^{13}C NMR ($CDCl_3$): δ 178.35, 165.37, 164.59 (C═O), 162.469–115.58 (C═C, aromatic and aliphatic), 81.36 (N—C—N), 57.53, 57.28

(CH_3O). Analysis calculated for $C_{38}H_{29}N_3O_5$: C 75.12, H 4.77, N 6.91%; found: C 74.75, H 4.83, N 7.01%.

Crystal data

$C_{38}H_{29}N_3O_5$	D_x = 1.348 Mg m^{-3}
M_r = 607.64	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2924 reflections
a = 13.196 (2) Å	θ = 4.5–50.6°
b = 19.853 (3) Å	μ = 0.09 mm^{-1}
c = 11.9850 (19) Å	T = 150 (2) K
β = 107.470 (3)°	Block, yellow
V = 2995.0 (8) Å^3	0.47 × 0.29 × 0.29 mm
Z = 4	

Data collection

Bruker SMART1000 diffractometer	3696 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.092
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	θ_{max} = 27.5°
T_{min} = 0.959, T_{max} = 0.974	$h = -16 \rightarrow 17$
25141 measured reflections	$k = -25 \rightarrow 24$
6764 independent reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.2194P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.058	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.139	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 1.01	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
6764 reflections	
415 parameters	
H-atom parameters constrained	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12A···O2^{i}	0.95	2.32	3.163 (3)	148
C20—H20A···O4^{ii}	0.95	2.43	3.366 (3)	169

Symmetry codes: (i) $1-x, 1-y, 2-z$; (ii) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

H atoms were positioned geometrically and refined with a riding model (including torsional freedom for methyl groups), with C—H = 0.95–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) [1.5U_{eq}(C) for methyl groups].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

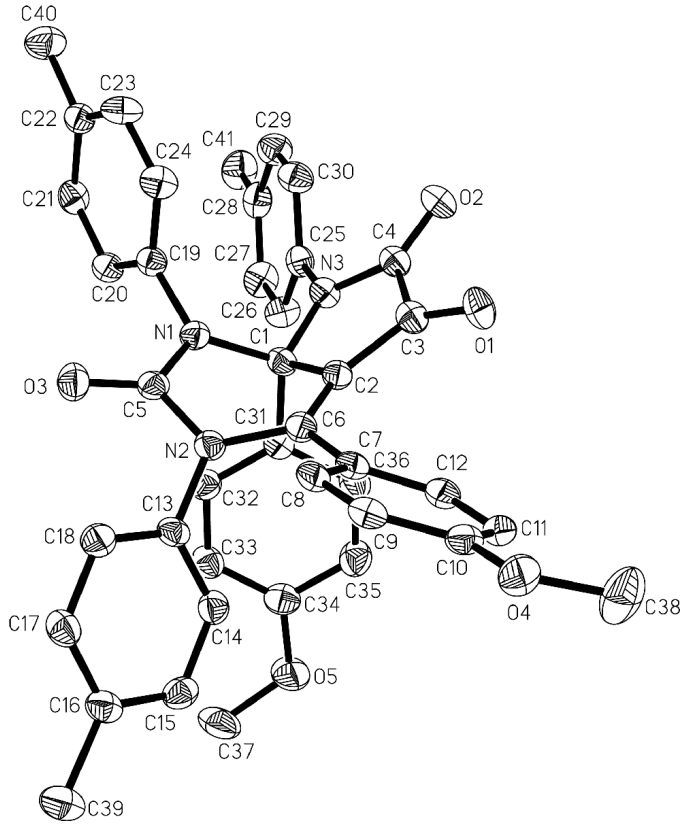


Figure 1
A view of the molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

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