

Harry Adams,^{a*} Samuel M. Hawxwell,^a Mustafa Saçmacı,^b Şevket Hakan Üngören,^b Yunus Akçamur^b and Recep Şahingöz^c^aDepartment of Chemistry, University of Sheffield, Brook Hill, Sheffield S3 7HF, England,^bDepartment of Chemistry, Yozgat Faculty of Arts and Sciences, Erciyes University, 66200-Yozgat, Turkey, and ^cDepartment of Physics, Yozgat Faculty of Arts and Sciences, Erciyes University, 66200-Yozgat, TurkeyCorrespondence e-mail:
h.adams@sheffield.ac.uk

Key indicators

Single-crystal X-ray study
T = 150 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.050
wR factor = 0.129
Data-to-parameter ratio = 16.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

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

The structure of the title compound, $\text{C}_{41}\text{H}_{35}\text{N}_3\text{O}_5$, is stabilized by intra- and intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

Pyrrolo[2,3]pyrimidines are an important class of compounds that are structurally and chemically related to nucleosides and some antibiotics (Ohgi *et al.*, 1979; Tolmann *et al.*, 1968). The well known biological activity of these compounds has led to intensive investigation of their use as antitumor, anti-allergic, antiviral and anti-inflammatory agents (Hutzenlaub *et al.*, 1972; Smith *et al.*, 1972).

In the light of this, we have synthesized and characterized the title compound, (2), and have determined its structure by X-ray analysis.

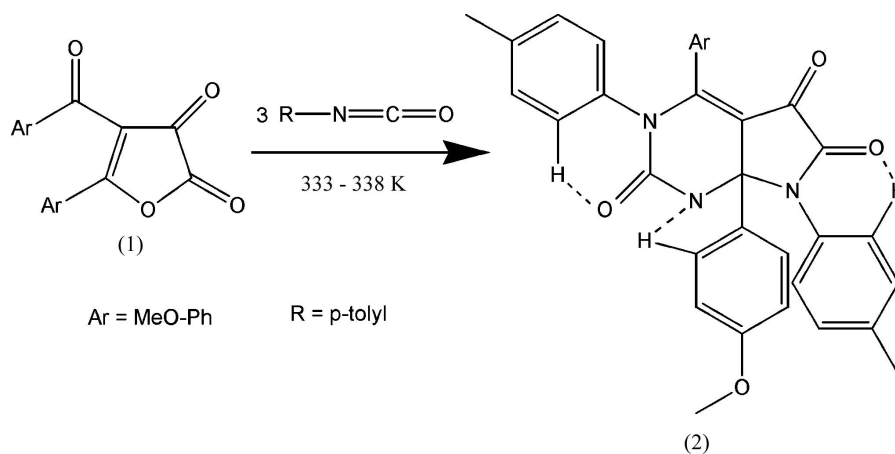


Fig. 1 shows the molecular structure of (2), with the atomic numbering scheme. The six-membered non-aromatic ring (C2/C1/N1/C5/N2/C6) has a total puckering amplitude of 0.532 (3) Å (Cremer & Pople, 1975) and a boat conformation [$\varphi = 179.5$ (4)° and $\theta = 105.0$ (5)°]. The five-membered aromatic ring (C1–C4/N3) and the five benzene rings (C7–C12, C13–C18, C19–C24, C25–C30 and C31–C36) are each essentially planar, with r.m.s. deviations of 0.0296 (13), 0.0104 (14), 0.0069 (15), 0.0067 (15), 0.046 (16) and 0.126 (15) Å, respectively. The bond lengths and angles are in agreement with reported literature values (Allen *et al.*, 1987).

The structure is stabilized by intra- and intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1). In the crystal structure, the $\text{C}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds link the molecules into dimers which are stacked along the *b* axis (Fig. 2).

Experimental

Compound (1) was prepared from the cyclocondensation reaction that occurs between *p,p'*-dimethoxydibenzoylketene and oxalyl

chloride (Hökelek *et al.*, 2002). Compound (2) was obtained from (1) (1.0 g, 2.96 mmol) by reaction with an excess of *p*-tolyl isocyanate in a 25 ml round-bottomed flask equipped with a calcium chloride tube. The mixture was heated at 338 K for 24 h. After cooling to room temperature, the residue was triturated with anhydrous diethyl ether, and the crude product was recrystallized from ethanol (yield 1.26 g, 66%; m.p. 475 K). IR (KBr, cm^{-1}): ν 1727 (C3–O1), 1709 (C4–O2),

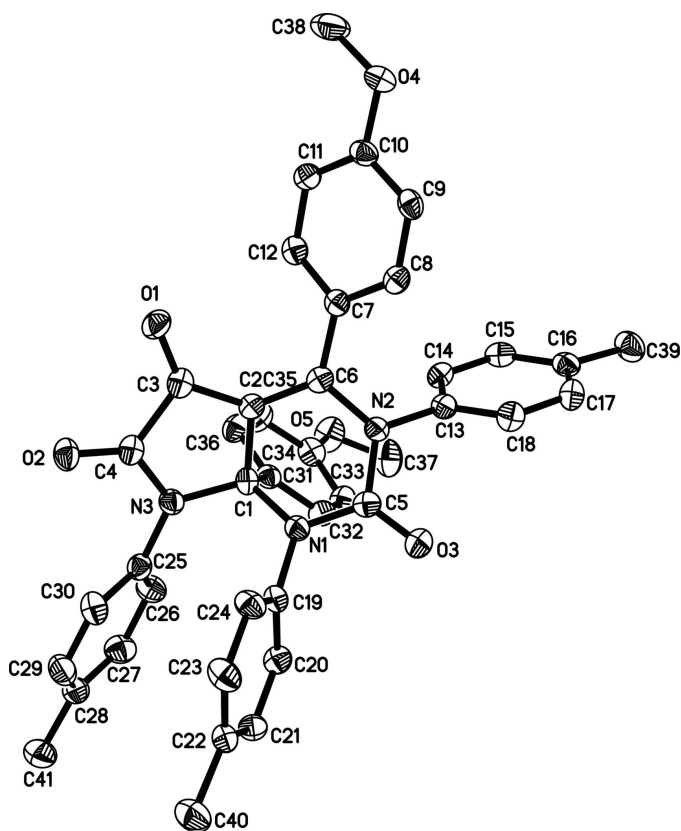


Figure 1
The molecular structure of (2), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

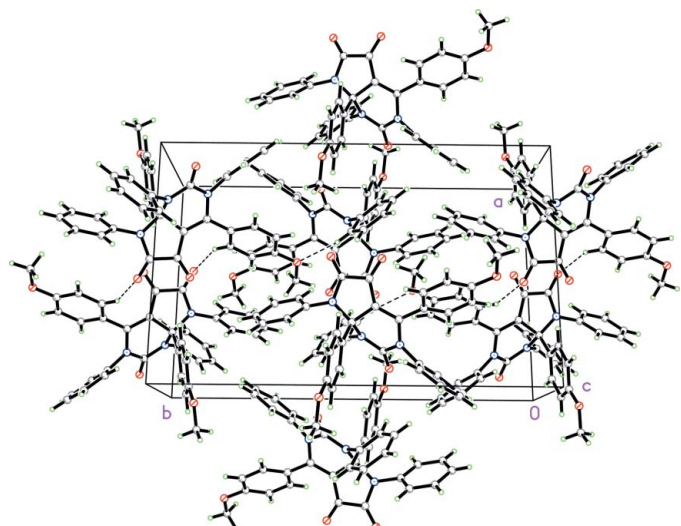


Figure 2
Packing diagram of (2); C–H...O hydrogen bonds are shown as dashed lines.

1684 (C5–O3); ^1H NMR (CDCl_3 , p.p.m.): δ 7.64–6.04 (*m*, 20H, Ar–H), 3.88, 3.77 (*s*, 6H, CH_3O), 2.25, 2.18, 2.15 (*s*, 9H, Ar– CH_3); ^{13}C NMR (CDCl_3 , p.p.m.): δ 178.51 (C3–O1), 165.58 (C4–O2), 164.47 (C5–O3), 162.35–115.52 (C=C, arom. and aliph.), 81.36 (N1–C1–N2), 57.50 (O4–C38), 57.28 (O5–C37), 23.00, 22.94, 22.92 (Ar– CH_3). Analysis calculated for $\text{C}_{41}\text{H}_{35}\text{N}_3\text{O}_5$: C 75.80, H 5.39, N 6.47%; found: C 76.02, H 5.69, N 6.22%.

Crystal data

$\text{C}_{41}\text{H}_{35}\text{N}_3\text{O}_5$
 $M_r = 649.72$
 Triclinic, $P\bar{1}$
 $a = 10.1797$ (12) Å
 $b = 12.5347$ (14) Å
 $c = 13.6855$ (16) Å
 $\alpha = 107.999$ (2)°
 $\beta = 99.985$ (2)°
 $\gamma = 90.504$ (2)°
 $V = 1632.0$ (3) Å³
 $Z = 2$
 $D_x = 1.322$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3062 reflections
 $\theta = 4.7$ – 50.5 °
 $\mu = 0.09$ mm⁻¹
 $T = 150$ (2) K
 Block, yellow
 $0.38 \times 0.21 \times 0.10$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\text{min}} = 0.968$, $T_{\text{max}} = 0.991$
 18940 measured reflections
 7331 independent reflections
 4028 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.054$
 $\theta_{\text{max}} = 27.6$ °
 $h = -13 \rightarrow 13$
 $k = -16 \rightarrow 16$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.129$
 $S = 0.98$
 7331 reflections
 445 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.1342P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

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

D–H...A	D–H	H...A	D...A	D–H...A
C9–H9A...O1 ⁱ	0.95	2.55	3.409 (3)	150
C11–H11A...O2 ⁱⁱ	0.95	2.53	3.158 (3)	123
C18–H18A...O3	0.95	2.58	2.927 (3)	102
C30–H30A...O2	0.95	2.55	2.928 (3)	104
C32–H32A...N1	0.95	2.51	2.858 (3)	102
C39–H39C...O5 ⁱⁱⁱ	0.98	2.48	3.422 (3)	160
C40–H40C...O4 ^{iv}	0.98	2.54	3.341 (3)	139

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

H atoms were positioned geometrically [0.95 (CH) and 0.98 Å (CH_3)] and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H})$ values of 1.2 (1.5 for methyl) times U_{eq} .

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SHELXTL (Bruker, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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