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Key indicators

Single-crystal X-ray study
 $T = 193$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.044
 wR factor = 0.120
Data-to-parameter ratio = 13.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.3-(1*H*-Indol-3-yl)-4-(3,4,5-trimethoxyphenyl)-2,5-dihydro-1*H*-pyrrole-2,5-dioneThe crystal structure of the title compound, $\text{C}_{21}\text{H}_{18}\text{N}_2\text{O}_5$, was determined in order to study the electrocyclic reactivity of 3,4-diaryl-1*H*-pyrrole-2,5-dione derivatives. Intermolecular hydrogen bonds form sheets.

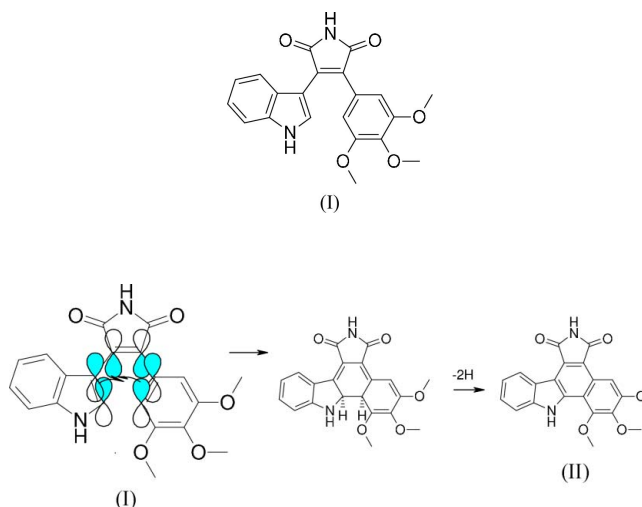
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Comment

The title compound, (I), containing the maleimide (1*H*-pyrrole-2,5-dione) moiety as the core structure, was prepared in an attempt to synthesize analogues of combretastatin A-4, a well known antithrombotic compound (Woods *et al.*, 1995). Among the synthetic methods used for preparing asymmetrical 3,4-diarylmaleimides, the method of Faul *et al.* (1999) has been used. Purification was achieved by column chromatography, yielding (I) as yellow crystals. Crystals of (I) suitable for X-ray analysis precipitated at 278 K from an ethanol solution by slow evaporation; however, at room temperature in an ethyl acetate solution, compound (I) was found to be chemically unstable and generated the corresponding carbazole, (II), which is described in a subsequent report (Peifer *et al.*, 2005). The reaction scheme below shows the disrotatory cyclization of (I) and subsequent oxidation to yield (II).



A comparable mechanism for the reactivity of the class of 3,4-diarylmaleimides has been reported by Sanchez-Martinez *et al.* (2003) and Harris *et al.* (1993). The constitutions of maleimide (I) and carbazole (II) were investigated by ¹H and ¹³C NMR spectroscopic analyses and were confirmed by X-ray analysis, showing the compounds to have the desired structures (Fig. 1). We now report the X-ray crystal structure analysis which definitively proves the structure of (I) and confirms the assumption of oxidative cyclization reactivity of

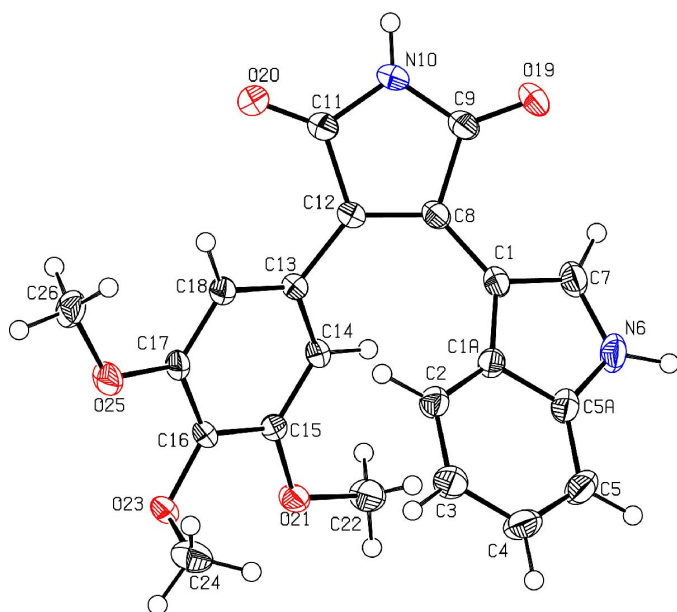


Figure 1
ORTEP view (Johnson, 1976) of a molecule of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are depicted as circles of arbitrary size.

(I). Intermolecular N—H···O hydrogen bonds form sheets parallel to the *ab* plane (see Table 1 and Fig. 2).

Experimental

See *Comment* and Faul *et al.* (1999) for details. The title compound was obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{21}H_{18}N_2O_5$
 $M_r = 378.38$
Monoclinic, $C2/c$
 $a = 18.448$ (2) Å
 $b = 9.9631$ (4) Å
 $c = 20.029$ (2) Å
 $\beta = 108.422$ (5)°
 $V = 3492.7$ (5) Å³
 $Z = 8$

$D_x = 1.439$ Mg m⁻³
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
 $\theta = 65$ –73°
 $\mu = 0.86$ mm⁻¹
 $T = 193$ (2) K
Block, yellow
0.65 × 0.60 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
Absorption correction: ψ scan (CORINC; Dräger & Gattow, 1971)
 $T_{\min} = 0.647$, $T_{\max} = 0.772$
3673 measured reflections
3559 independent reflections

3465 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$
 $\theta_{\text{max}} = 74.3^\circ$
 $h = 0 \rightarrow 22$
 $k = 0 \rightarrow 12$
 $l = -25 \rightarrow 23$
3 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.121$
 $S = 1.10$
3559 reflections
268 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 3.5777P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.00211 (15)

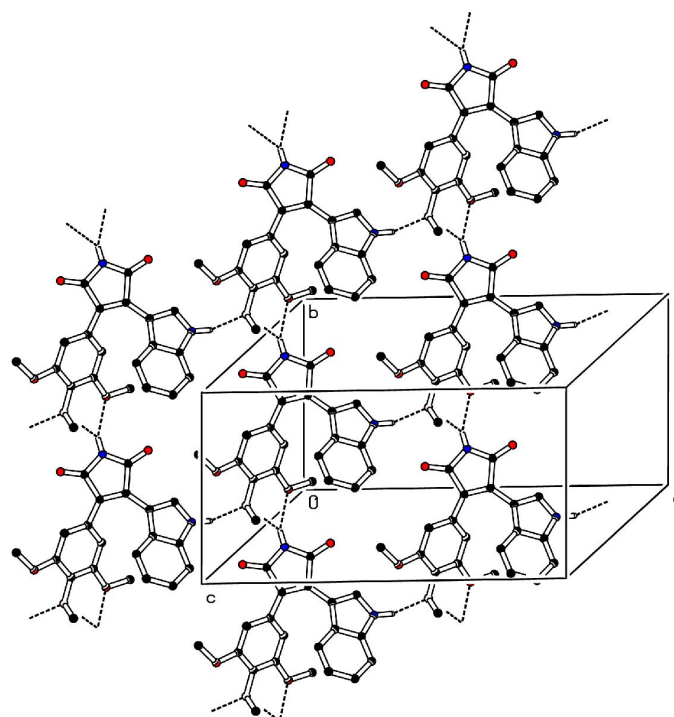


Figure 2
Part of the packing diagram of (I). Dashed lines indicate hydrogen bonds. Only important H atoms are shown.

Table 1

Hydrogen-bonding 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>
N6—H6···O23 ⁱ	0.96	2.03	2.9005 (17)	150
N10—H10···O21 ⁱⁱ	0.88	2.13	2.9320 (17)	152
N10—H10···O23 ⁱⁱ	0.88	2.60	3.2759 (18)	134

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

All H atoms were located in difference Fourier maps. The range of C—H distances is 0.89–1.01 Å and N—H distances is 0.88–0.96 Å. The U_{iso} values were refined freely (one per H atom and one per methyl group). The methyl H atoms were allowed to ride but not to rotate.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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