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5,6,7-Trimethoxy-2,3-dihydro-1*H*,8*H*-benzo[*a*]-pyrrolo[3,4-*c*]carbazole-1,3-dione dimethyl sulfoxide solvate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.076
wR factor = 0.187
Data-to-parameter ratio = 14.2

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

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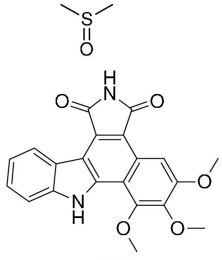
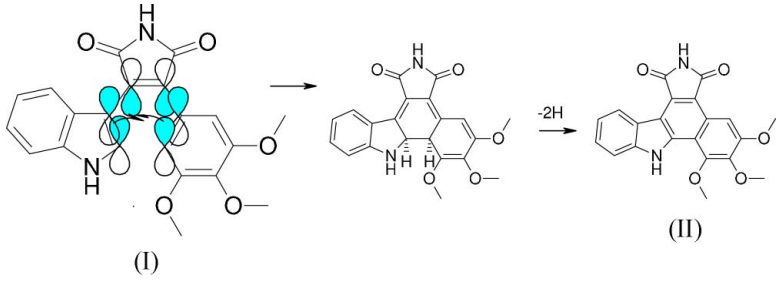
The crystal structure of the title compound, $C_{21}H_{16}N_2O_5\cdot C_2H_6OS$, was determined to investigate the electrocyclic reactivity of 3,4-diaryl-1*H*-pyrrole-2,5-diones (3,4-bisarylmaleimides) to the yield corresponding carbazole derivatives.

Comment

The title compound, (III), bearing the carbazole moiety as a core structure, was accidentally isolated from an ethyl acetate solution of 3-(indol-3-yl)-4-(3,4,5-trimethoxyphenyl)-1*H*-pyrrole-2,5-dione, (I) (Peifer *et al.*, 2005) at room temperature. The reaction scheme below shows the disrotatory cyclization of (I) and subsequent oxidation to yield (II).



(III)



(I) (II)

The analytically pure 1*H*-pyrrole-2,5-dione derivative was found to undergo a reaction (monitored by thin-layer chromatography) producing (II). A comparable mechanism of reactions of the class of 1*H*-pyrrole-2,5-diones had been reported by Sanchez-Martinez *et al.* (2003) and Harris *et al.* (1993). However, after 24 h in an ethyl acetate solution, approximately 10% of (II) could be determined by high-performance liquid chromatographic analysis. Compound (II) was subsequently isolated by column chromatography and found to be chemically stable. Crystals of (I) precipitated at 278 K from DMSO. We now report the X-ray crystal structure analysis of carbazole (III), which is the DMSO solvate of carbazole (II) and which confirms the structure and strongly supports the mechanism of oxidative cyclization of the 1*H*-

pyrrole-2,5-dione derivative to generate compound (II). The solvent DMSO molecule is linked *via* a hydrogen bond to the carbazole molecule (see Table 1 and Fig. 1).

Experimental

The title compound was obtained by crystallization of a DMSO solution of (II).

Crystal data

$C_{21}H_{16}N_2O_5\cdot C_2H_6OS$
M_r = 454.50
Monoclinic, $P2_1/c$
a = 7.994 (2) Å
b = 20.040 (4) Å
c = 13.644 (4) Å
β = 106.586 (12)°
V = 2094.8 (9) Å^3
Z = 4
D_x = 1.441 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 30–44°
μ = 1.76 mm^{-1}
T = 295 (2) K
Needle, yellow
0.24 × 0.06 × 0.04 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*CORINC*; Dräger & Gattow, 1971)
T_{min} = 0.783, T_{max} = 0.932
4534 measured reflections
4228 independent reflections
2943 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 74.0°
$h = -9 \rightarrow 0$
$k = 0 \rightarrow 24$
$l = -16 \rightarrow 17$
3 standard reflections
frequency: 60 min
intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.187
S = 1.11
4228 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 1.9378P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···O21	0.79	2.20	2.741 (4)	126
N10—H10···O2*L*	0.87	2.00	2.849 (5)	164

H atoms attached to N were located in a difference map and refined isotropically. Other H atoms were placed at calculated positions and refined with fixed isotropic displacement parameters using a riding model [C—H = 0.93 or 0.96 Å and U(H) = 1.2 or 1.5 times U_{eq}(C); the methyl groups were allowed to rotate but not to tip.

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*.

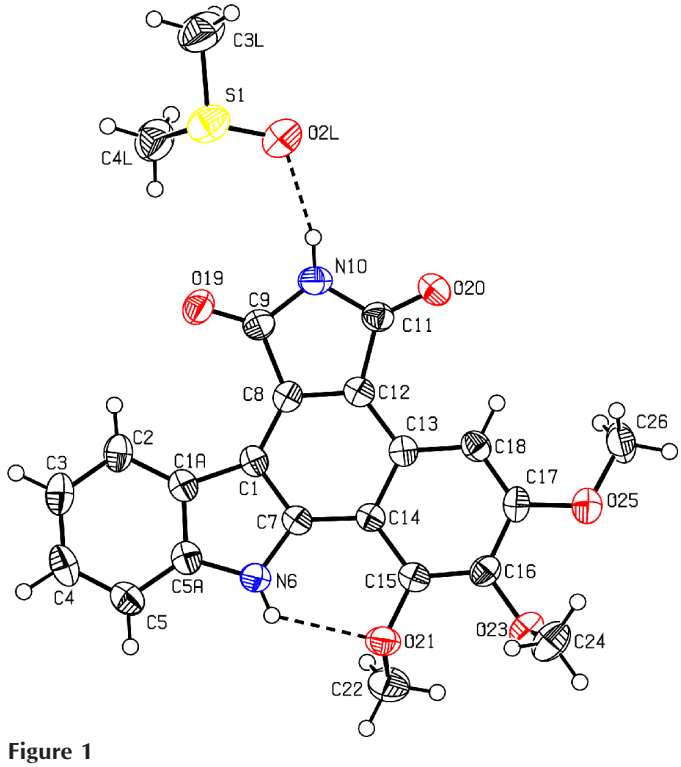


Figure 1
ORTEPII view (Johnson, 1976) view of (III). Displacement ellipsoids are shown at the 50% probability level. H atoms are depicted as circles of arbitrary size. Dashed lines indicate hydrogen bonds.

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