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

D—H···A	D—H	H···A	D···A	D—H···A
O3—HO3···O2	0.80	2.35	2.728 (4)	110
O2—HO2···O4^{i}	0.89	1.92	2.791 (3)	169
O3—HO3···O1^{ii}	0.80	2.18	2.918 (4)	154

Symmetry codes: (i) $\frac{3}{2} - x, y - \frac{1}{2}, -z$; (ii) $x, y - 1, z$.

The hydroxyl and amide H atoms were determined from a difference Fourier map. All H atoms were refined as riding with a common atomic displacement parameter of 0.076 Å^2.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *XCAD*4 (Harms, 1997). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* and *XPMA* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*93.

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The twisted structure of 9-(4-cyanophenyl)carbazole

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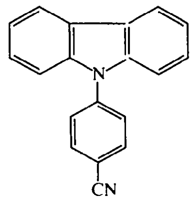
Abstract

The crystal structure determination of the title compound, $C_{19}H_{12}N_2$, has been undertaken with a view to understanding the nature of the fluorescence band of the system. There is a significant twist between the cyanophenyl and carbazolyl moieties.

Comment

A number of electron donor–acceptor (EDA) molecules exhibit, in addition to the locally excited (LE) fluorescence, a second anomalous fluorescence band at longer wavelength that originates from a twisted intramolecular charge-transfer (TICT) state in which the donor and acceptor moieties are mutually orthogonal to each other (Rotkiewicz *et al.*, 1973; Rettig, 1986). An alternative mechanism in terms of a change in the pyramidalization of the amino N atom has been proposed recently to account for this phenomenon (Il'ichev *et al.*, 1998).

The title compound, (I), displays a single fluorescence band that has been tentatively assigned as an emission from the TICT state (Rettig & Zander, 1982). Since the ground-state structure of a system often determines the excited-state conformation of the molecule, we have determined the crystal structure of this system.



(I)

The molecule (I) exhibits a torsion angle C12—N1—C13—C18 of 48.3 (2)° (Fig. 1). This observation suggests that the electron donor (carbazolyl) and acceptor (cyanophenyl) moieties are twisted with respect to each

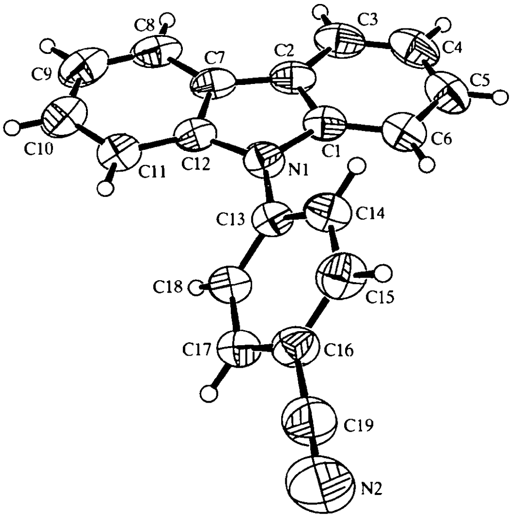


Fig. 1. A view of the molecular structure of (I) showing the atom labels. Displacement ellipsoids are shown at the 40% probability level.

other in the ground state. Since it is generally assumed that if an EDA molecule exists in a twisted conformation in the ground state, the emission is likely to take place from a twisted geometry, it is quite likely that the single Stokes-shifted fluorescence band that is observed for the present system originates from the TICT state.

Experimental

The title compound was prepared according to a general procedure (Bridger *et al.*, 1968). A mixture of carbazole (1 g, 3 mmol) and sodium hydride (72 mg, 3 mmol) was stirred in dry dimethylformamide (15 ml) under a nitrogen atmosphere for 2 h. The sodium salt of carbazole so formed was then heated at 393 K with 4-fluorobenzonitrile (0.363 g, 3 mmol) and sodium iodide (0.456 g, 6 mmol) for about 20 h. The product, along with unreacted reactants, was precipitated by adding water to the reaction mixture. The precipitate was dried and the title compound was separated by column chromatography on a silica-gel column. A mixture of ethyl acetate and hexane (50:50) was used as the eluent. Colourless crystals were obtained from absolute ethanol upon slow evaporation of the solvent.

Crystal data

$C_{19}H_{12}N_2$
$M_r = 268.31$
Monoclinic
$P2_1/c$
$a = 8.2739\,(12)$ Å
$b = 20.681\,(4)$ Å
$c = 8.5913\,(11)$ Å
$\beta = 104.937\,(12)°$
$V = 1420.4\,(4)$ Å^3
$Z = 4$
$D_x = 1.255$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 1.5–27.5°$
$\mu = 0.075$ mm^{-1}
$T = 293\,(2)$ K
Block
1.04 × 0.72 × 0.72 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
3457 measured reflections
3251 independent reflections
2043 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.016$
$\theta_{\rm max} = 27.48°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 26$
$l = -11 \rightarrow 10$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.115$
$S = 1.046$
2043 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0610P)^2 + 0.9105P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.12$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.034 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *Xtal*3.4 (Hall *et al.*, 1995). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEX* (McArdle, 1995). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1320). Services for accessing these data are described at the back of the journal.

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Nitro derivatives of glutethimide

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Abstract

Crystallographic experiments performed on single crystals of *m*-nitroglutethimide [3-ethyl-3-(3′-nitrophenyl)piperidine-2,6-dione, $C_{13}H_{14}N_2O_4$], (I), and *o,p*-