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Key indicators
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.112
Data-to-parameter ratio = 13.3

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

The title compound, C_{23}H_{13}N_{5}, derived from cinnamyl alcohol and 2,2′-(isoindolin-1,3-diyldiene)bispropanedinitrile, is a heterocyclic TCNQ analogue of interest as an electron-deficient component in charge-transfer complexes. A small perturbation of the four C=C=N angles from linearity is observed, which are in the range 173.41 (18)–176.3 (2)°; the C≡N bond lengths are in the range 1.144 (2)–1.146 (2) Å. The terminal phenyl group is oriented at an angle of 77.17 (6)° to the C\textsubscript{6}N ring and the C≡C bond is short, 1.319 (2) Å. There are no classical hydrogen bonds, although intramolecular C–H···N and intermolecular C–H···π(arene) interactions influence the crystal-structure packing.

Comment

Organic conductors are currently an important research area in materials chemistry (Martin et al., 1997; Yamashita & Tomura, 1998), with special interest in the interaction of π-deficient and π-excessive materials in 1:1 complexes, e.g. TCNQ/TTF, where TCNQ is tetracyanoquinodimethane and TTF is tetrathiafulvalene.

![Diagram of the title compound](image)

The bond lengths and angles in the heterocyclic ring of the title compound, (I) (Fig. 1), are similar to those reported previously in related systems (McNab et al., 1997; Brady et al., 1998; Gallagher & Murphy, 1999; Brady & Gallagher, 2000). For TCNQ (tetracyanoquinodimethane) systems (280 examples), the average exocyclic C\textsubscript{sp}^2–C\textsubscript{sp}^2 and C\textsubscript{sp}^2–C\textsubscript{sp}^1 bond lengths are 1.392 (17) and 1.427 (10) Å, respectively; thus, in (I), the C\textsubscript{6}A–C\textsubscript{7}A, C\textsubscript{6}A–C\textsubscript{8}A, and C\textsubscript{6}B–C\textsubscript{7}B and C\textsubscript{6}B–C\textsubscript{8}B bond lengths in the range 1.425 (3)–1.436 (3) Å are normal (Orpen et al., 1994). The four nitrile C≡N values are from 1.144 (2) to 1.146 (2) Å, which compare with the average C≡N dimension from the literature, 1.144 (8) Å (Orpen et al., 1994). The exocyclic indolinyl ring C=C bond lengths are 1.372 (2) and 1.374 (2) Å, which are longer than typical double bonds. The four remaining exocyclic indolinyl cyano C=C bond lengths are in the range 1.425 (3)–1.436 (3) Å and similar.
to previously reported values. The angles which the C(C≡N)₂ groups make with the C₃N ring are 7.01 (10) (C₆A) and 2.33 (10)° (C₆B), demonstrating a small twist from planarity about the C₄—C₆(A/B) bonds.

The hydrogen bonding in (I) is dominated by intramolecular C—H···N interactions and close contacts (details in Table 2). There are two intramolecular C(arene)—H···π(C≡N) interactions with C12—H12···N2A and C15—H15···N2B C···N distances of 3.390 (3) and 3.390 (2) Å, respectively. A C1—H1B···N3A intramolecular contact is present [C1···N3A 3.401 (2) Å and C—H···N 139°]. A C—H···π(arene) interaction is also present, C14—H14···Cg1, where Cg1 is the centroid of [C21—C26] [symmetry code: (i) x/2, y, z; details in Table 2]. The closest C14···C26 contact distance is 3.696 (2) Å with a C14—H14···Cg1 angle of 121°.

### Experimental

The title compound was prepared by the 5 d reaction under argon of DIAD (diisopropylazodicarboxylate) (0.37 g, 1.9 mmol), triphenylphosphine (0.49 g, 1.9 mmol), 2,2’-(isodolin-1,3-diylidene)bispropanedinitril (0.25 g, 1.9 mmol) (Farbenfabriken Bayer Aktiengesellschaft, 1968), cinnamyl alcohol (0.25 g, 1.9 mmol) in 40 ml tetrahydrofuran. 2,2’-(Cinnamylisoindolin-1,3-diylidene)bispropanedinitril, (I), was isolated as an orange crystalline material after column chromatography (m.p. 469–471 K uncorrected). IR (KBr, cm⁻¹): 3049, 2372, 2335, 2224, 1563, 1465, 1407, 1228, 1146, 1109, 983, 775, 723; UV–Vis (CH₂CN) λmax(ε): 552 (5250), 459 (6667), 414 (38333), 392 (39167), 249 (47083) nm; ¹H NMR (400 MHz, δ, CDCl₃): 8.75 (m, 2H, aromatic), 7.85 (m, 2H, aromatic), 7.37 (m, 2H, aromatic), 7.30 (m, 3H, aromatic); 6.50 (d, J = 16 Hz, 1H), 6.25 (m, 1H), 5.50 (m, 2H); ¹³C NMR (δC, CDCl₃): 158.7, 135.0, 134.8, 134.0, 130.9, 128.8, 128.7, 126.9, 126.0, 120.5, 113.1, 112.3, 62.1, 47.8.

### Crystal data

C₂₃H₂₃N₅

Mᵡ = 359.38

Monoclinic, P2₁/n

a = 14.3872 (18) Å

b = 8.2696 (10) Å

c = 16.1442 (15) Å

β = 108.327 (7)°

V = 1823.4 (4) Å³

Z = 4

\[ D_r = 1.309 \text{ Mg m}^{-3} \]

Mo Kα radiation

Cell parameters from 33 reflections

\[ \theta = 7.1°–20.5° \]

\[ \mu = 0.08 \text{ mm}^{-1} \]

\[ T = 296 (1) \text{ K} \]

Block, orange

0.43 × 0.38 × 0.28 mm

### Data collection

Bruker P4 diffractometer

\[ h = -1 \rightarrow 17 \]

\[ k = -1 \rightarrow 10 \]

4331 measured reflections

2630 reflections with \( I > 2\sigma(I) \)

\[ R_{int} = 0.012 \]

\[ \theta_{max} = 25.5° \]

### Refinement

Refinement on \( F^2 \)

\[ R(F^2) = 0.042 \]

\[ wR(F^2) = 0.112 \]

\[ S = 1.06 \]

3367 reflections

253 parameters

H-atom parameters constrained

### Table 1

Selected geometric parameters (Å, °).

| N1—C1 | 1.469 (2) | C1—C2 | 1.495 (2) |
| C1—C2 | 1.388 (2) | C2—C3 | 1.319 (2) |
| N1—C5 | 1.388 (2) | C5—C6 | 1.471 (2) |
| C1—C7A | 1.145 (2) | C4—C6A | 1.374 (2) |
| N3A—C8A | 1.146 (2) | C4—C11 | 1.460 (2) |
| N2B—C7B | 1.144 (2) | C5—C6B | 1.372 (2) |
| N3B—C8B | 1.143 (2) | C5—C16 | 1.461 (2) |
| C1—N1—C4 | 125.99 (12) | N1—C5—C6B | 125.80 (14) |
| C1—N1—C5 | 123.11 (12) | N1—C4—C11 | 107.12 (12) |
| C4—N1—C5 | 110.82 (12) | N1—C5—C16 | 106.81 (12) |
| C1—N1—C2 | 113.84 (13) | N2A—C7A—C6A | 176.3 (2) |
| C1—C2—C3 | 126.34 (15) | N3A—C8A—C6B | 175.3 (2) |
| C2—C3—C2 | 126.82 (16) | N2B—C7B—C6B | 175.81 (18) |
| N1—C4—C6A | 121.02 (15) | N3B—C8B—C6B | 173.41 (18) |
| C4—N1—C1—C2 | −102.16 (17) | C1—C2—C3—C21 | −174.28 (16) |
| C5—N1—C1—C2 | 81.50 (18) | C2—C3—C2—C22 | −1.5 (3) |
| N1—C1—C2—C3 | 6.6 (2) | C2—C3—C2—C26 | 176.66 (17) |

### Table 2

Hydrogen-bonding geometry (Å, °).

Cg1 is the centroid of [C21—C26].

| D—H···A | D—H | H···A | D···A | D—H···A |
| C1—H1B···N3A | 0.97 | 2.61 | 3.401 (2) | 139 |
| C1—H12···N2A | 0.93 | 2.59 | 3.390 (3) | 145 |
| C1—H12···C7A | 0.93 | 2.45 | 3.015 (2) | 119 |
| C1—H15···N2B | 0.93 | 2.59 | 3.390 (2) | 145 |
| C1—H15···C7B | 0.93 | 2.46 | 3.015 (2) | 119 |
| C1—H14···Cg1 | 0.93 | 3.20 | 3.984 (2) | 143 |

Symmetry code: (i) \( x, y, z \).


Colm Crean et al. • C₂₃H₂₃N₅ o237
In (I), all H atoms bound to C atoms were treated as riding, with $U_{eq}(H) = 1.5U_{eq}(C)$ for methylene H atoms and $1.2U_{eq}(C)$ for the remainder. Examination of the structure with PLATON (Spek, 1998) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: XSCANS (Bruker, 1994); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: NRCVAX96 (Gabe et al., 1989) and SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPIII (Burnett & Johnson, 1996) and PLATON (Spek, 1998); software used to prepare material for publication: NRCVAX96, SHELXL97 and WordPerfect macro PREP8 (Ferguson, 1998).

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References