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2,3:9,10-Dibenzotricyclo[5.3.0.0^{4,8}]deca-2,5,9-triene-6,7-dicarbonitrile

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Key indicators

Single-crystal X-ray study
T = 178 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.099
Data-to-parameter ratio = 13.0

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

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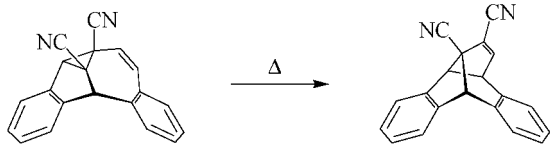
In the title compound, $C_{20}H_{12}N_2$, the fused ring system leads to angle strain (*e.g.* sp^3 angles of *ca* 98° or *ca* 130°). The molecules are linked by one C—H···N and two C—H···π interactions to form layers perpendicular to [10$\bar{1}$].

Comment

Recently, we described the crystal structure of 9,10-dicyanodibenzoisobullvalene [(1); Jones *et al.*, 2003]. Since the polycyclic carbon skeleton of (1) contains a vinylcyclopropane subunit, which, in principle, can undergo a ring-opening reaction, we decided to pyrolyze (1) (Witulski, 1992). We describe here the structure of the main isomerization product, (2).



(1) (2)

The molecule of (2) is shown in Fig. 1. The strain imposed by the fused-ring system is apparent in, for example, the lengthened single bond C1—C7, the narrow sp^2 angles C4—C5—C6/C5—C6—C7 and sp^3 angles C1—C7—C8/C4—C8—C7, and the widened sp^3 angles C8—C9—C15/C1—C10—C18 (Table 1).

The molecules are linked to form ribbons parallel to the *b* axis by a weak C—H···N hydrogen bond (Table 2) and then, more strikingly, crosslinked by two C—H···π interactions to the centroid (cent) of the ring C2/C3/C11–C14, to form layers parallel to (101); C5—H5···cent, with H···cent = 2.55 Å and C—H···cent = 153° for the operator 1 − *x*, *y*, $\frac{1}{2}$ − *z*, and C16—H16···cent, with H···cent = 2.58 Å and C—H···cent = 155°

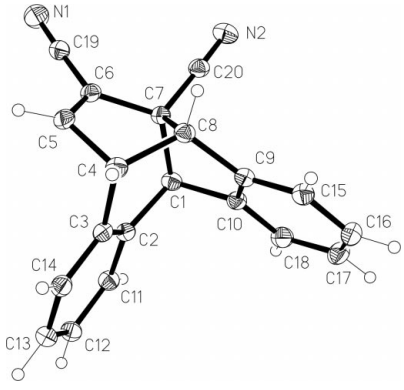


Figure 1
The molecule of compound (2) in the crystal. The H atom at C1 is eclipsed. Ellipsoids are drawn at the 30% probability level and H-atom radii are arbitrary.

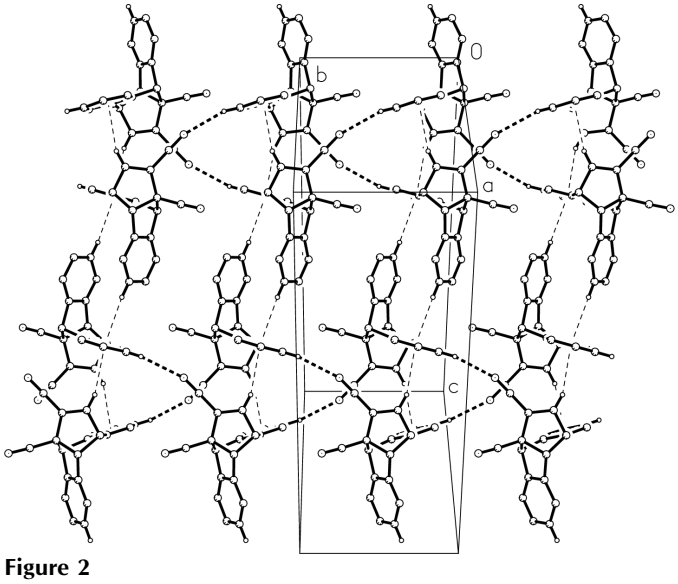


Figure 2
Packing diagram of compound (2). Hydrogen bonds of the type C—H···N are shown as thick dashed lines and C—H···π interactions as thin dashed lines. H atoms not involved in hydrogen bonds have been omitted. The view direction is perpendicular to $(10\bar{1})$, with the rings that accept the C—H···π interactions being viewed edge-on.

for the operator $\frac{3}{2}-x, \frac{3}{2}-y, -z$. The C—H distances were normalized to 1.08 Å to calculate these values. A packing diagram is shown in Fig. 2.

Experimental

A toluene solution of (1) was heated at 503 K for 24 h in a sealed ampoule, leading to two isomerization products in the ratio 9:1. These were separated by thick-layer chromatography and the major product [(2); 10% yield] was recrystallized from chloroform/pentane (Witulski, 1992).

Crystal data

$C_{20}H_{12}N_2$
$M_r = 280.32$
Monoclinic, $C2/c$
$a = 15.834\ (5)$ Å
$b = 8.196\ (3)$ Å
$c = 22.849\ (7)$ Å
$\beta = 99.11\ (3)^\circ$
$V = 2927.8\ (17)$ Å^3
$Z = 8$
$D_x = 1.272$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
$\theta = 10$–11.5°
$\mu = 0.08$ mm^{-1}
$T = 178\ (2)$ K
Prism, colourless
0.7 × 0.4 × 0.4 mm

Data collection

Nicolet *R*3 diffractometer
ω scans
2674 measured reflections
2596 independent reflections
1938 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.1^\circ$
$h = -18 \rightarrow 18$
$k = -9 \rightarrow 1$
$l = -27 \rightarrow 0$
3 standard reflections
every 147 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.099$
$S = 1.05$
2596 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0374P)^2 + 2.4161P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0026 (4)

Table 1
Selected geometric parameters (Å, °).

C1—C7	1.578 (2)	C5—C6	1.331 (2)
C6—C5—C4	109.66 (15)	C4—C8—C7	98.28 (13)
C5—C6—C7	109.81 (15)	C15—C9—C8	130.24 (16)
C8—C7—C1	98.99 (13)	C18—C10—C1	130.13 (16)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···N1^{i}	0.95	2.59	3.314 (3)	134

Symmetry code: (i) $1-x, 1+y, \frac{1}{2}-z$.

H atoms were included using a riding model, with fixed C—H bond lengths (sp^2 C—H = 0.95 Å and methine C—H = 1.00 Å); U_{iso}(H) values were fixed at $1.2U_{eq}$ of the parent atom.

Data collection: *P*3 (Nicolet, 1987); cell refinement: *P*3; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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