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9,10-Dicyanodibenzoisobullvalene

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Key indicators

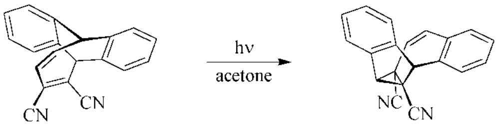
Single-crystal X-ray study
T = 178 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.111
Data-to-parameter ratio = 12.8

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

In the title compound, $C_{20}H_{12}N_2$, the bond lengths in the cyclopropane ring are 1.526, 1.532 and 1.541 (2) Å. The molecules are linked by two C—H···N contacts.

Comment

Prebullvalene, bicyclo[4.2.2]deca-2,4,7,9-tetraene, is a $(CH)_{10}$ hydrocarbon whose photo-isomerization behaviour has been investigated (for a review see Scott & Jones, 1972). Our studies of the addition of cyanoacetylene to aromatic compounds (Hopf & Witulski, 1995) furnished us with the dibenzoprebullvalenedicarbonitrile, (1), and we have investigated its photochemical behaviour in acetone solution with light of wavelength > 300 nm. The photo-isomerization yielded the title compound (2), and we present here its crystal structure.



(1) (2)

Whereas the parent hydrocarbon prebullvalene isomerizes to bullvalene when irradiated (Scott & Jones, 1972; Jones & Scott, 1967), the photorearrangement of (1) takes a completely different course. To rationalize the formation of (2), we propose that the process is initiated by a [1,3] carbon shift followed by a di-π-methane rearrangement.

The structure of (2) is shown in Fig. 1. Molecular dimensions are normal. Bond lengths in the cyclopropane ring (Table 1) are somewhat greater than the average value of

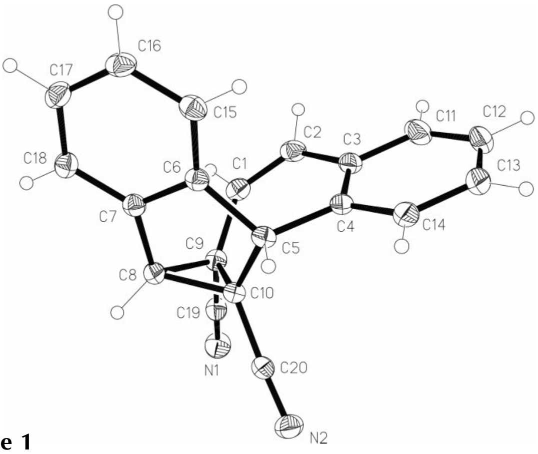


Figure 1
The molecule of compound (2) in the crystal. Displacement ellipsoids are drawn at the 30% probability level and H-atom radii are arbitrary.

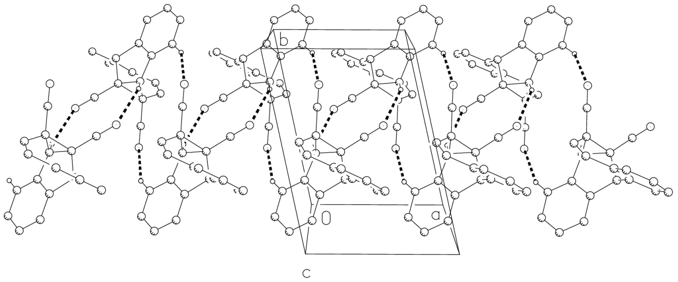


Figure 2
The packing of compound (2) in the crystal. Dashed lines indicate hydrogen bonds; only those H atoms participating in hydrogen bonds are included. The view direction is perpendicular to the *ab* plane.

1.509 Å established by Allen (1980); see also Rozsondai (1995).

The molecules are linked to form columns parallel to the *a* axis by two C—H···N interactions (Fig. 2 and Table 2).

Experimental

Compound (1) was irradiated for 4 h at room temperature using a 450 W mercury high-pressure lamp, forming (2) in 65% yield (Witulski, 1992). The product was crystallized from chloroform/pentane.

Crystal data

$C_{20}H_{12}N_2$
$M_r = 280.32$
Triclinic, $P\overline{1}$
$a = 6.722\ (3)$ Å
$b = 9.154\ (3)$ Å
$c = 12.143\ (4)$ Å
$\alpha = 96.85\ (3)^\circ$
$\beta = 90.65\ (3)^\circ$
$\gamma = 102.51\ (3)^\circ$
$V = 723.7\ (5)$ Å^3

$Z = 2$
$D_x = 1.286$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
$\theta = 10$–12°
$\mu = 0.08$ mm^{-1}
$T = 178\ (2)$ K
Prism, colourless
$0.7 \times 0.4 \times 0.2$ mm

Data collection

Nicolet *R*3 diffractometer
ω scans
Absorption correction: none
3368 measured reflections
2539 independent reflections
1923 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$

$\theta_{\rm max} = 25.0^\circ$
$h = -7 \rightarrow 2$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$
3 standard reflections every 147 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.03$
2539 reflections
199 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.0943P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.14$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C8—C10	1.526 (2)	C9—C10	1.532 (2)
C8—C9	1.541 (2)		
C10—C8—C9	59.92 (10)	C8—C10—C9	60.54 (10)
C10—C9—C8	59.54 (10)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18···N1$^{\rm i}$	0.95	2.65	3.526 (2)	154
C8—H8···N2$^{\rm ii}$	1.00	2.57	3.440 (2)	145

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1-x, 1-y, -z$.

H atoms were included using a riding model with fixed C—H bond lengths (Csp^2—H = 0.95 Å and methine C—H = 1.00 Å); $U_{\rm iso}$(H) values were fixed at $1.2U_{\rm eq}$ 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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