Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(3,4),(6,7)Dibenzo-9,10-dicyanotriquinacene

Peter G. Jones,[a]* Peter Bubenitschek,[b] Henning Hopf[b] and Bernhard Witulski[b]

[a]Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and [b]Institut für Organische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany

Correspondence e-mail: p.jones@tu-bs.de

Received 3 March 2003
Accepted 4 March 2003
Online 31 March 2003

Key indicators

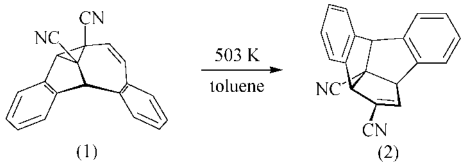
Single-crystal X-ray study
T = 178 K
Mean σ(C–C) = 0.005 Å
R factor = 0.065
wR factor = 0.216
Data-to-parameter ratio = 12.8

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

The title compound, $C_{20}H_{12}N_2$, displays slightly lengthened C—C bonds at the central C atom of the triquinacene framework (mean 1.572 Å) and wide exocyclic angles at the benzo-annelation sites (*ca* 127°). The packing is determined by two C—H···N contacts and one of the type C—H···π.

Comment

Dibenzoisobullvalene dicarbonitrile [(1); Jones *et al.*, 2003], readily available by photoisomerization of the corresponding dibenzoprebullvalene isomer (Hopf & Witulski, 1995), contains a vinylcyclopropane subunit that, in principle, can undergo thermal ring expansion to a five-membered ring. To test this possibility, we heated (1) in toluene in a sealed ampoule at 503 K. From the product mixture, the ring-expanded dinitrile (2), a derivative of dibenzotriquinacene, can indeed be isolated in good yield (86%). The structure of (2) was previously elucidated by its spectroscopic and analytical data (Witulski, 1992) and the X-ray structural analysis is reported here.



The molecular structure of (2) is shown in Fig. 1. Bond lengths and angles may be regarded as normal; however, some deviations from standard values are observed, *e.g.* the slightly lengthened bonds to the central atom C10 of the triquinacene framework, and the widened exocyclic angles at the benzo-annelation sites (Table 1).

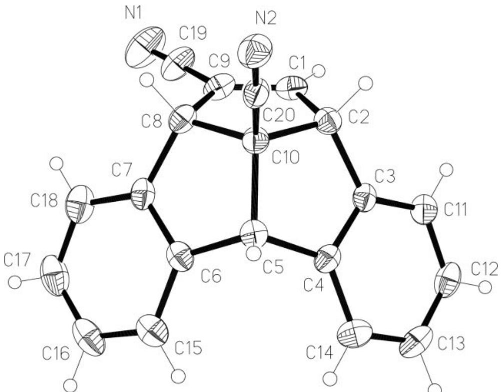


Figure 1
The molecular structure of compound (2) in the crystal. Displacement ellipsoids are shown at the 30% probability level and H-atom radii are arbitrary.

DOI: 10.1107/S1600536803005221

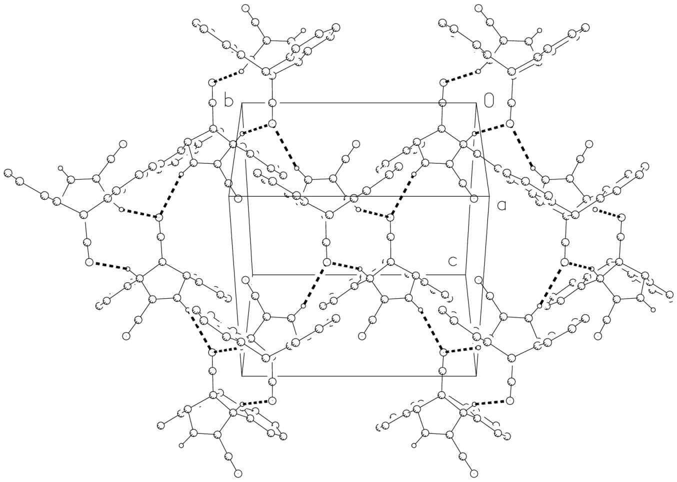


Figure 2
Packing diagram of compound (2), viewed perpendicular to the $(10\overline{1})$ plane. Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonds have been omitted.

The molecules are linked to form corrugated layers parallel to (101) by two C—H···N contacts (Table 2). Additionally, the centroid (Cent) of ring C6/C7/C15–C18 is involved in a contact of the type C—H···π, *viz*. C17—H17···Cent($1 - x$, $1 - y$, $-z$), with H···Cent = 2.72 Å and an angle of 150°. This contact, not shown in Fig. 2, links the layers to complete the three-dimensional packing.

Experimental

The title compound was prepared according to the method of Witulski (1992) and recrystallized from chloroform/pentane.

Crystal data

$C_{20}H_{12}N_2$
$M_r = 280.32$
Monoclinic, $P2_1/n$
$a = 9.528$ (4) Å
$b = 12.401$ (6) Å
$c = 12.265$ (6) Å
$\beta = 97.73$ (4)°
$V = 1436.0$ (12) Å^3
$Z = 4$

$D_x = 1.297$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 49 reflections
$\theta = 10$–11.5°
$\mu = 0.08$ mm^{-1}
$T = 178$ (2) K
Prism, colourless
0.50 × 0.35 × 0.25 mm

Data collection

Nicolet *R*3 diffractometer
ω scans
Absorption correction: none
4770 measured reflections
2542 independent reflections
1266 reflections with $I > 2\,s(I)$
$R_{\rm int} = 0.053$

$\theta_{\rm max} = 25.1$°
$h = -4 \rightarrow 11$
$k = -13 \rightarrow 14$
$l = -14 \rightarrow 14$
3 standard reflections every 147 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.217$
$S = 1.04$
2542 reflections
199 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.4194P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.22$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—C10	1.564 (5)	C8—C10	1.574 (5)
C5—C10	1.577 (5)		
C11—C3—C2	127.0 (3)	C15—C6—C5	127.1 (4)
C14—C4—C5	127.4 (4)	C18—C7—C8	127.2 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···N2$^{\rm i}$	0.95	2.67	3.478 (5)	144
C8—H8···N2$^{\rm ii}$	1.00	2.59	3.528 (5)	157

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

H atoms were included using a riding model with fixed C—H bond lengths (Csp^2—H = 0.95 Å and methine = 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.

Financial support from the Fonds der Chemischen Industrie is gratefully acknowledged. We thank Mr A. Weinkauf for technical assistance.

References

Hopf, H. & Witulski, B. (1995). *Modern Acetylene Chemistry*, edited by P. J. Stang and F. Diederich, pp. 33–66. Weinheim, Germany: VCH Verlagsgesellschaft.
Jones, P. G., Hopf, H. & Witulski, B. (2003). *Acta Cryst.* E**59**, o169–o170.
Nicolet (1987). *P*3 and *XDISK*. Nicolet Instrument Corporation, Madison, USA.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Witulski, B. (1992). PhD Thesis, Technical University of Braunschweig, Germany.