Crystal data

$C_{20}H_{24}N_4$
$M_r = 320.441$
Triclinic
$P\bar{1}$
$a = 8.4511\,(13)$ Å
$b = 9.8471\,(16)$ Å
$c = 12.2407\,(18)$ Å
$\alpha = 111.352\,(12)°$
$\beta = 91.970\,(14)°$
$\gamma = 108.781\,(11)°$
$V = 884.9\,(6)$ Å^3
$Z = 2$
$D_x = 1.202$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 16.48–18.07°$
$\mu = 0.068$ mm^{-1}
$T = 295$ K
Irregular
0.50 × 0.30 × 0.25 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3481 measured reflections
3147 independent reflections
2020 reflections with $I > 3\sigma(I)$

$R_{\rm int} = 0.009$
$\theta_{\rm max} = 26.32°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F
$R = 0.046$
$wR = 0.057$
$S = 1.49$
2020 reflections
217 parameters
H atoms: see below
Weighting scheme: see below

$(\Delta/\sigma)_{\rm max} = 0.0001$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.35$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

N1—C1	1.415 (4)	N3—C9	1.420 (4)
N1—C7	1.455 (5)	N4—C2	1.430 (4)
N1—C8	1.452 (5)	N4—C3	1.459 (4)
N2—C2	1.440 (4)	N4—C15	1.388 (4)
N2—C4	1.475 (4)	C1—C2	1.341 (4)
N2—C5	1.484 (4)	C3—C4	1.533 (5)
N3—C1	1.412 (4)	C6—C7	1.497 (5)
N3—C6	1.472 (4)		
C1—N1—C7	105.5 (3)	C2—N4—C15	125.7 (3)
C1—N1—C8	120.2 (3)	C3—N4—C15	121.3 (3)
C7—N1—C8	113.6 (3)	N1—C1—N3	107.6 (3)
C2—N2—C4	100.4 (3)	N1—C1—C2	127.1 (3)
C2—N2—C5	111.4 (3)	N3—C1—C2	125.0 (3)
C4—N2—C5	111.6 (3)	N2—C2—N4	108.5 (3)
C1—N3—C6	109.0 (3)	N2—C2—C1	123.7 (3)
C1—N3—C9	123.3 (3)	N4—C2—C1	125.1 (3)
C6—N3—C9	115.9 (3)	N4—C3—C4	102.5 (3)
C2—N4—C3	108.1 (3)	N4—C15—C16	121.8 (3)

The weighting scheme used was w = (threshold/F^2) if $F \geq$ threshold or $w = 0$ if $F^2 <$ cutoff.σF^2 (threshold = 93.03 and cutoff = 4). All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 0.95 Å from their parent atoms. After refining the methyl H atoms for a few cycles, a riding model was used for all H atoms, with $B_{\rm iso}({\rm H}) = 1.3B_{\rm eq}({\rm C})$.

Data collection: *CAD*-4 *Express* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIR* (Burla *et al.*, 1989) in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP* (Johnson, 1965) in *MolEN*.

The authors acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: BM1050). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). **C53**, 241–243

9,10-Bis{2-[4-(*N*,*N*-diphenylamino)phenyl]ethynyl}anthracene

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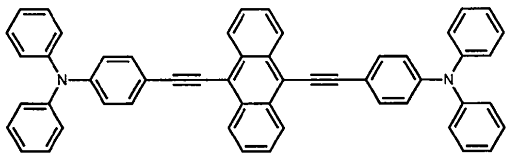
Abstract

The crystal structure of the title compound, $C_{54}H_{36}N_2$, shows that the anthracene rings in the unit cell do not stack in a face-to-face manner. Instead, the anthracene

rings are positioned over the planar N atom of the triphenylamino group.

Comment

Organic materials containing the triphenylamine moiety are of considerable interest for their technological applications as photoconductors and hole transport layers (Junji, 1994). We have prepared a novel class of compounds based on triphenylamine and anthracene units. Knowledge of the intermolecular interactions is necessary in order to understand the solid-state electronic and transport properties of these compounds and so a structural study of the title compound, (I), was undertaken.



(I)

The crystal structure of (I) shows that the anthracene core units in the unit cell do not stack face-to-face, but instead overlap with the planar N atom of the triphenylamine segment (Figs. 1 and 2). The center of the anthracene ring lies at a distance of 4.59 Å from each N atom at an offset angle of 11.2°.

Experimental

The title compound was synthesized according to known literature procedures and was crystallized from $CDCl_3$ by slow evaporation.

Crystal data

$C_{54}H_{36}N_2$
$M_r = 712.85$
Monoclinic
$P2_1/n$
$a = 11.582\,(2)$ Å
$b = 9.407\,(2)$ Å
$c = 18.429\,(4)$ Å
$\beta = 108.14\,(2)°$
$V = 1908.1\,(7)$ Å^3
$Z = 2$
$D_x = 1.241$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.36–9.94°$
$\mu = 0.072$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.40 × 0.32 × 0.30 mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–θ scan profiles
Absorption correction: by integration
$T_{min} = 0.986$, $T_{max} = 0.997$
2958 measured reflections
2802 independent reflections
2042 reflections with $I > 2\sigma(I)$

$R_{int} = 0.0284$
$\theta_{max} = 23.47°$
$h = -12 \rightarrow 0$
$k = 0 \rightarrow 10$
$l = -19 \rightarrow 20$
3 standard reflections
frequency: 90 min
intensity decay: 0.09%

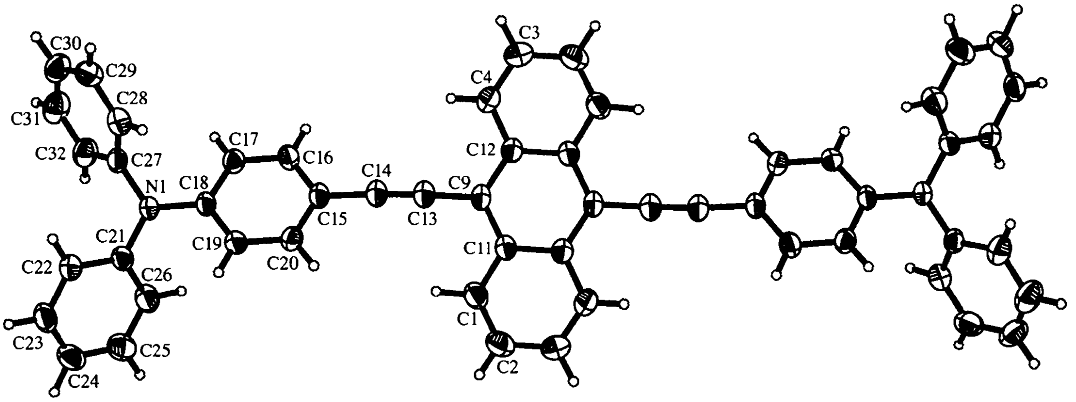


Fig. 1. *ORTEPII* (Johnson, 1971) plot showing 50% probability ellipsoids for non-H atoms and H atoms as circles of arbitrary size.

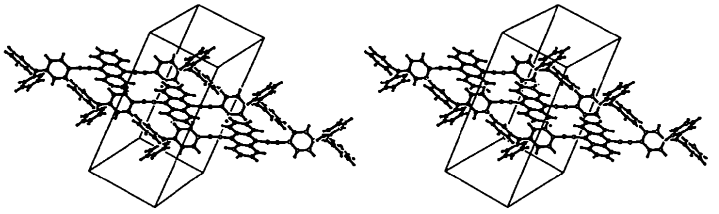


Fig. 2. A stereoview showing the packing of (I). Note that the anthracene rings do not exhibit π–π stacking.

Refinement

Refinement on F^2
$R(F) = 0.0438$
$wR(F^2) = 0.1228$
$S = 1.040$
2802 reflections
308 parameters
H-atom coordinates refined, $U_{\rm eq}$(H) constrained to a single free variable and refined
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 0.3764P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.131$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.141$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C18	1.403 (2)	C15—C20	1.395 (3)
N1—C21	1.423 (3)	C16—C17	1.371 (3)
N1—C27	1.430 (3)	C17—C18	1.391 (3)
C1—C2	1.352 (4)	C18—C19	1.385 (3)
C1—C11	1.422 (3)	C19—C20	1.375 (3)
C2—C3	1.404 (4)	C21—C22	1.380 (3)
C3—C4	1.354 (4)	C21—C26	1.383 (3)
C3—C2	1.404 (4)	C22—C23	1.384 (3)
C4—C12	1.418 (3)	C23—C24	1.354 (4)
C9—C11	1.410 (3)	C24—C25	1.370 (4)
C9—C12	1.413 (3)	C25—C26	1.379 (3)
C9—C13	1.433 (3)	C27—C28	1.375 (3)
C11—C12	1.426 (3)	C27—C32	1.377 (3)
C12—C11	1.426 (3)	C28—C29	1.390 (4)
C13—C14	1.192 (3)	C29—C30	1.374 (4)
C14—C15	1.435 (3)	C30—C31	1.352 (4)
C15—C16	1.386 (3)	C31—C32	1.365 (4)
C18—N1—C21	121.6 (2)	C19—C18—N1	120.9 (2)
C18—N1—C27	119.0 (2)	C17—C18—N1	121.2 (2)
C21—N1—C27	119.3 (2)		

The red transparent prismatic data crystal was mounted using epoxy resin on a thin glass fiber with the $(\bar{1}0\bar{1})$ scattering planes roughly normal to the spindle axis. The data crystal was bound by the $(1\bar{1}\bar{2})$, $(\bar{1}12)$, $(11\bar{2})$, $(\bar{1}\bar{1}2)$, $(\bar{1}01)$ and $(10\bar{1})$ faces.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software* and *PROFILE* (Blessing, Coppens & Becker, 1972). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1971). Software used to prepare material for publication: *CIFTAB* in *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: BK1186). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Novel Tricyclic Cyclobutanone Ketal

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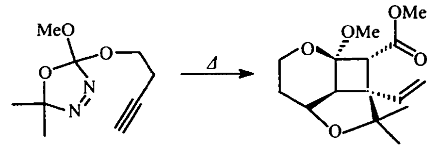
Abstract

Methyl (±)-(1α,1aα,3aα,6aα,6bα)-1a-ethenyl-1,1a,2,3a,-4,5,6a,6b-octahydro-6a-methoxy-2,2-dimethyl-3,6-dioxacyclobut[*cd*]indene-1-carboxylate, $C_{15}H_{22}O_5$, adopts a hemispherical conformation with the methoxy, methoxycarbonyl and vinyl groups pointing outwards from the hemisphere. The tetrahydropyran ring is in a chair conformation and the tetrahydrofuran ring is in an envelope conformation.

Comment

The title compound, (2), is formed in high yield by a complex cascade of reactions resulting from thermolysis of oxadiazoline, (1), in benzene (Kassam & Warkentin, 1994). Its complexity, including a tricyclic framework and five asymmetric centres, left several structural possibilities on the basis of spectroscopic evidence alone and so an X-ray structure determination was necessary.



(1) (2)

The molecule consists of a four-membered carbon ring, a tetrahydrofuran ring and a tetrahydropyran ring, all with a common CH group (C6b). The cavity formed is enclosed by the O3 atom, the C5 methylene group