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10-Phenyl-10-phenylethynyl-9-methylenefluorene

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.076
Data-to-parameter ratio = 12.9

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

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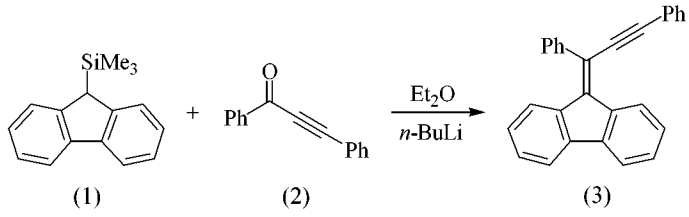
In the title compound [systematic name: 9-(1,3-diphenylprop-2-ynylidene)fluorene], $C_{28}H_{18}$, the two halves of the fluorene moiety subtend an angle of 7.84 (9)°; the interplanar angle about the exocyclic double bond is 12.3 (1)°. The packing involves three C—H···π contacts.

Comment

In connection with our studies of the thermal isomerization of 1,3-hexadien-5-ynes [a summary of such reactions leading to benzene derivatives is presented by Zimmermann (2001)], we needed a sample of 9-(1,3-diphenylprop-2-ynylidene)fluorene, (3), for comparison. The compound was prepared as described in the *Experimental* section, and characterized by spectroscopic data and the structure determination described here.



(1) (2) (3)

The molecule is shown in Fig. 1. Molecular dimensions may be regarded as normal. The two halves of the fluorene moiety subtend an interplanar angle of 7.84 (9)°. The orientation of the two phenyl rings is defined by the torsion angles C9—C10—C19—C20 62.9 (2)° and C9—C10···C13—C14 −22.3 (2)°, and the rotation about the C9═C10 bond by the angle of 12.3 (1)° between the planes C1A,C8A,C9,C10 and C9,C10,C11,C19.

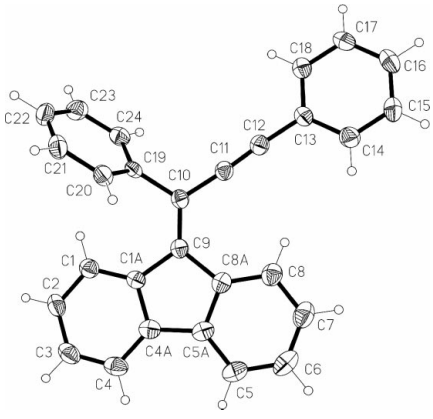


Figure 1
The molecule of the title compound in the crystal structure. Displacement ellipsoids are drawn at the 50% probability level. H-atom radii are arbitrary.

The following short but markedly non-linear contacts are observed from various hydrogen atoms to the centroids of the rings C1,C1A,C2,C3,C4,C4A (Cent1) and C19–24 (Cent2) (C—H normalized to 1.08 Å): H24···Cent1 (H···Cent 2.60 Å, angle at H 147°, operator of Cent x, $1+y$, z); H4···Cent1 (2.71 Å, 138°, $-x, y-\frac{1}{2}, \frac{1}{2}-z$) and H16···Cent2 (2.70 Å, 134°, $1-x$, $2-y$, $1-z$). The acceptor of this last contact may alternatively be considered to be the midpoint of the bond C21—C22 (distance 2.67 Å, angle 160°).

Experimental

The title compound (3) was prepared in 48% yield by Peterson olefination of phenyl phenylethynyl ketone [1,3-diphenylpropyn-3-one, (2)] and 9-trimethylsilylfluorene (1) in diethyl ether, using *n*-butyllithium as a base (Berger, 2004). Single crystals were obtained by slow cooling of solutions of (3) in pentane.

Crystal data

$C_{28}H_{18}$	$D_x = 1.262$ Mg m^{-3}
$M_r = 354.42$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 62 reflections
$a = 14.0901$ (14) Å	$\theta = 3.5$–10°
$b = 5.8367$ (8) Å	$\mu = 0.07$ mm^{-1}
$c = 23.519$ (2) Å	$T = 173$ (2) K
$\beta = 105.383$ (6)°	Prism, yellow
$V = 1864.9$ (4) Å^3	0.70 × 0.40 × 0.15 mm
$Z = 4$	

Data collection

Siemens *P*4 diffractometer	$\theta_{max} = 25.0°$
ω scans	$h = -16 \rightarrow 2$
Absorption correction: none	$k = -6 \rightarrow 0$
3940 measured reflections	$l = -27 \rightarrow 27$
3266 independent reflections	3 standard reflections
1970 reflections with $I > 2\,s(I)$	every 247 reflections
$R_{int} = 0.026$	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0331P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.076$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.82$	$\Delta\rho_{max} = 0.12$ e Å^{-3}
3266 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
253 parameters	

Table 1
Selected geometric parameters (Å, °).

C9—C10	1.362 (2)	C11—C12	1.196 (2)
C1—C1A—C9	131.65 (15)	C12—C11—C10	176.30 (18)
C8—C8A—C9	131.71 (16)	C11—C12—C13	176.55 (18)
C1A—C9—C10—C11	−173.09 (15)	C1A—C9—C10—C19	11.1 (2)
C8A—C9—C10—C11	12.8 (3)	C8A—C9—C10—C19	−162.94 (15)

H atoms were included using a riding model with fixed C—H bond lengths of 0.95 Å; U(H) values were fixed at 1.2 times U_{eq} of the parent atom.

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; 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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References

Berger, H. (2004). PhD thesis. Technical University of Braunschweig, Germany.
Fait, J. (1991). *Manuals to X-ray program system*. Siemens X-ray Analytical Instruments Inc., Madison, Wisconsin, 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, Madison, Wisconsin, USA.
Zimmermann, G. (2001). *Eur. J. Org. Chem.* pp. 457–471.