

9-Methylene-10,10-bis(phenylethynyl)fluorene

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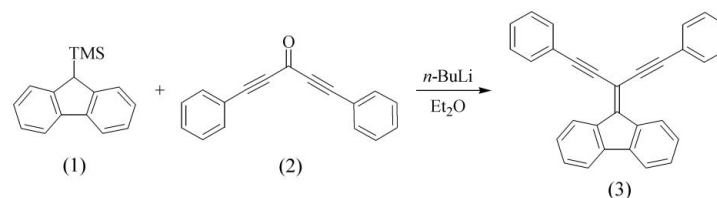
Key indicators

Single-crystal X-ray study
 $T = 173\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.045
 wR factor = 0.126
Data-to-parameter ratio = 7.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{30}\text{H}_{18}$, the fluorene moiety displays normal dimensions, such as a narrow ring angle of $105.3(3)^\circ$ at the 9-position, the atom bearing the exocyclic double bond. The phenyl rings subtend angles of $14.8(2)$ and $3.9(2)^\circ$ to the fluorene plane. Molecules are stacked parallel to the short c axis of $4.001(1)\text{ \AA}$.

Comment

In our studies concerning the reactivity (Eshdat *et al.*, 2002) and structural properties (Jones *et al.*, 2004) of cross-conjugated enediynes we have prepared title hydrocarbon (3). In this communication, we report the structural parameters of (3) as determined by single-crystal X-ray crystallography.



The molecule of (3) is shown in Fig. 1. Bond lengths and angles (*e.g.* the necessarily distorted, formally sp^2 angles at C9) may be regarded as normal (Table 1). The fluorene moiety is essentially planar (r.m.s. deviation of 13 atoms is 0.04 \AA), although a closer analysis reveals an interplanar angle of

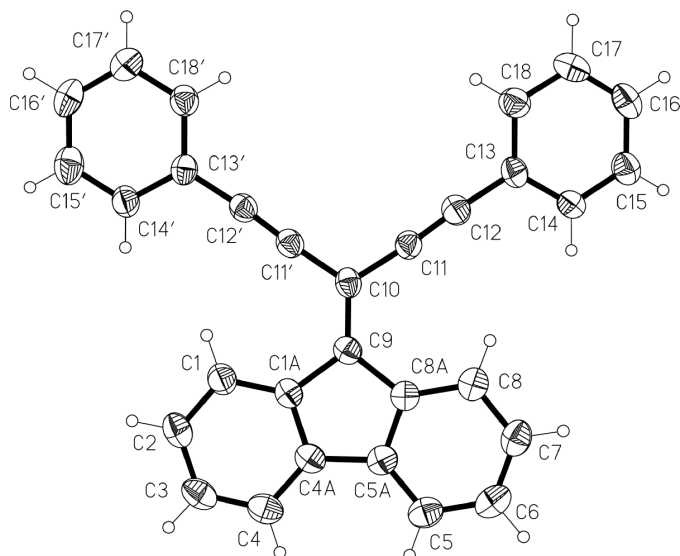


Figure 1
The molecule of (3) in the crystal structure. Displacement ellipsoids are shown at the 50% probability level. H atom radii are arbitrary.

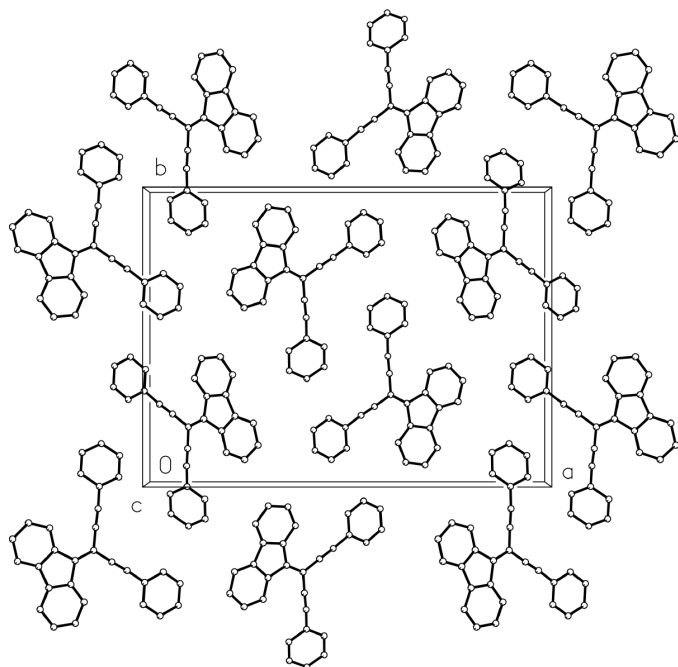


Figure 2
The crystal packing of (3), viewed parallel to the *c* axis. Radii are arbitrary. H atoms are omitted.

4.4 (2)° between its two six-membered rings. The torsion angles about the C9=C10 bond are close to the ideal values of 0/180°. The C13–C18 and C13'–C18' phenyl rings subtend interplanar angles of 14.8 (2) and 3.9 (2)°, respectively, to the overall fluorene plane.

The molecular packing (Fig. 2) shows no significant short contacts such as C–H... π interactions. The mean plane through the molecule that constitutes the asymmetric unit makes an angle of 34° to the *ab* plane. Molecules are necessarily stacked parallel to the short *c* axis.

Experimental

Compound (3) was prepared in 47% yield by the Peterson olefination of the diethynylketone (2) with 9-trimethylsilyl-fluorene, (1), using *n*-butyllithium as base in diethyl ether (Berger, 2005). All spectroscopic and analytical data agree with the structure given in the scheme. Recrystallization of (3) from chloroform/pentane afforded single crystals.

Crystal data

C₃₀H₁₈
 $M_r = 378.44$
 Orthorhombic, *Pna*2₁
 $a = 25.824$ (9) Å
 $b = 18.944$ (8) Å
 $c = 4.001$ (1) Å
 $V = 1957.3$ (12) Å³
 $Z = 4$
 $D_x = 1.284$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 50 reflections
 $\theta = 10$ –11.5°
 $\mu = 0.07$ mm⁻¹
 $T = 173$ (2) K
 Prism, yellow
 $0.64 \times 0.22 \times 0.18$ mm

Data collection

Siemens R3 diffractometer
 ω scans
 6350 measured reflections
 2004 independent reflections
 1320 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.066$
 $\theta_{\text{max}} = 25.1^\circ$

$h = -30 \rightarrow 30$
 $k = -22 \rightarrow 22$
 $l = 0 \rightarrow 4$
 3 standard reflections
 every 147 reflections
 intensity decay: 0%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.127$
 $S = 1.10$
 2004 reflections
 271 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0598P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C9–C10	1.367 (5)	C11'–C12'	1.198 (5)
C11–C12	1.197 (4)		
C1–C1A–C9	131.5 (3)	C9–C10–C11'	123.3 (3)
C4–C4A–C5A	130.4 (3)	C9–C10–C11	122.8 (3)
C5–C5A–C4A	130.4 (3)	C11'–C10–C11	113.9 (3)
C8–C8A–C9	131.2 (3)	C12–C11–C10	174.6 (4)
C10–C9–C8A	127.7 (3)	C11–C12–C13	175.8 (4)
C10–C9–C1A	126.9 (3)	C12'–C11'–C10	175.8 (4)
C8A–C9–C1A	105.3 (3)	C11'–C12'–C13'	175.1 (4)
C8A–C9–C10–C11'	177.9 (4)	C8A–C9–C10–C11	2.0 (6)
C1A–C9–C10–C11'	–0.4 (6)	C1A–C9–C10–C11	–176.3 (4)

H atoms were included using a riding model, starting from calculated positions, with C–H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. In the absence of significant anomalous scattering, Friedel opposite reflections were not measured, and the Flack (1983) parameter is therefore meaningless. To improve stability of refinement in view of the moderate data–parameter ratio, displacement parameters were subject to similarity and rigid-bond restraints.

Data collection: *P3* (Nicolet, 1987); cell refinement: *P3*; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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