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10,10-Bis(trimethylsilylethynyl)-9-methylenefluorene

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Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.005 Å
R factor = 0.060
wR factor = 0.137
Data-to-parameter ratio = 16.2

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

In the title compound {systematic name: 9-[bis(trimethylsilylethynyl)methylene]fluorene}, $C_{24}H_{26}Si_2$, the molecules are planar (except for the methyl groups) and pack in layers parallel to the bc plane at $x \simeq \frac{1}{4}$ and $\frac{3}{4}$. The mean distance between neighbouring layers, which are related by inversion, is 3.50 Å.

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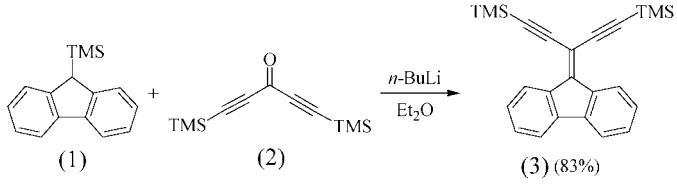
Comment

As we have recently shown (Eshdat *et al.*, 2002), 1,1-diethynylethenes (2-ethynylbut-1-en-3-ynes) undergo a novel cyclization to fulvalenes when subjected to reducing conditions (potassium in tetrahydrofuran). To generalize this process, we have prepared the fluorene derivative (3) (see *Experimental*), which was characterized by spectroscopic data and the structure determination described here.



The molecule is shown in Fig. 1. The molecular dimensions are normal. Except for the methyl groups, the molecule is essentially planar (r.m.s. deviation of non-H atoms is 0.02 Å).

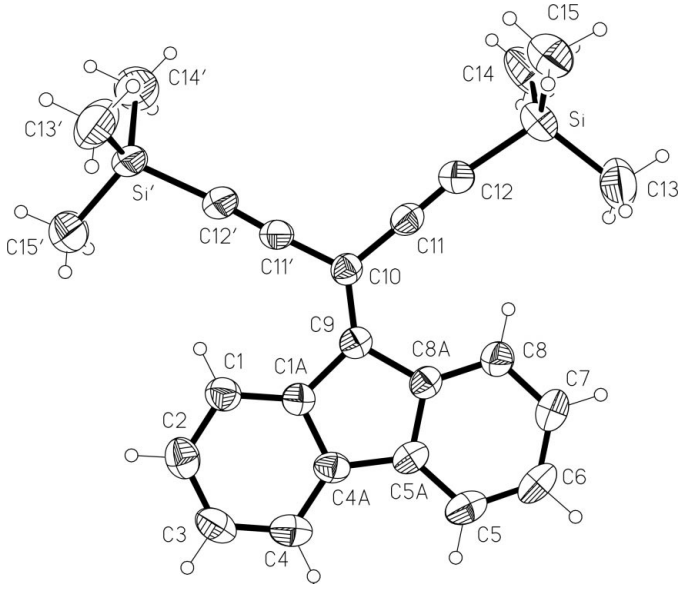


Figure 1
The molecule of (3). Displacement ellipsoids are drawn at the 50% probability level. H-atom radii are arbitrary.

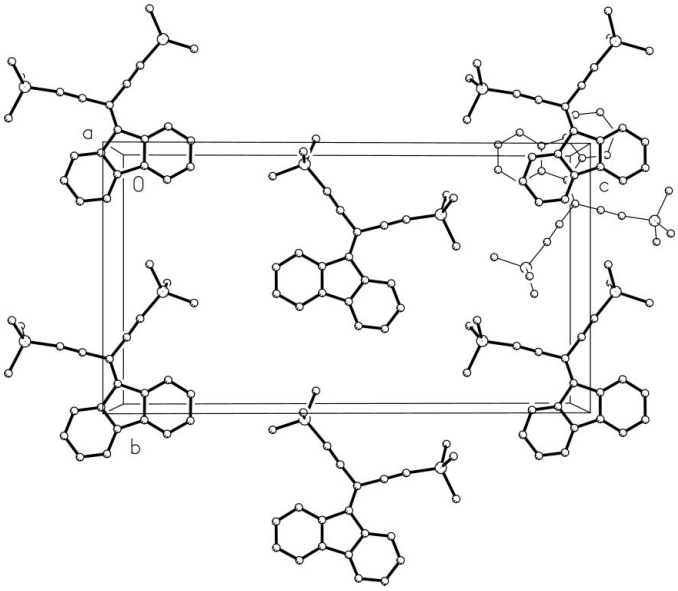


Figure 2
Packing diagram of (3), projected parallel to the *a* axis, showing one layer and (top right) the overlap with one molecule of the next layer, drawn with thinner bonds. H atoms have been omitted for clarity; radii are arbitrary.

The molecules pack in layers parallel to the *bc* plane, to which the molecular plane of the asymmetric unit makes an angle of 5.2°, at $x \simeq \frac{1}{4}$ and $\frac{3}{4}$ (Fig. 2). The mean distance between neighbouring layers, which are related by inversion, is $a/2$ = 3.50 Å. There are no unusually short intermolecular contacts.

Jones *et al.* (2004) describe a TCNQ adduct of the title compound.

Experimental

Compound (3) was prepared in 83% yield by Peterson olefination of the doubly protected ketone (2) with 9-trimethylsilylfluorene, (1), using *n*-butyllithium as base in diethyl ether (Berger, 2004). Single crystals were obtained by slow cooling of solutions of (3) in methanol.

Crystal data

$C_{24}H_{26}Si_2$
M_r = 370.63
Monoclinic, $P2_1/n$
a = 7.003 (2) Å
b = 13.219 (2) Å
c = 23.779 (4) Å
β = 91.64 (3)°
V = 2200.4 (8) Å^3
Z = 4
D_x = 1.119 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 48 reflections
θ = 10–11.5°
μ = 0.17 mm^{-1}
T = 203 (2) K
Tablet, yellow
0.4 × 0.4 × 0.2 mm

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
8747 measured reflections
3894 independent reflections
2212 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 25.1°
$h = -8 \rightarrow 8$
$k = -15 \rightarrow 0$
$l = -28 \rightarrow 28$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.137
S = 1.01
3894 reflections
241 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 0.8683P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C11—C12	1.194 (4)	C11′—C12′	1.192 (4)
C12—C11—C10	179.0 (4)	C12′—C11′—C10	176.9 (3)
C11—C12—Si	173.3 (3)	C11′—C12′—Si′	175.2 (3)

Methyl H atoms were located in difference syntheses, idealized (C—H = 0.98 Å and H—C—H = 109.5°) and treated as rigid groups allowed to rotate but not tip. Other H atoms were placed at calculated positions and included in the refinement as riding, with C—H bond lengths of 0.95 Å; U_{iso}(H) values were fixed at $1.2U_{eq}$ of the parent atom. Because the data were weak, restraints were applied to the displacement parameters of all non-H atoms and to the geometry (similarity of the two chemically equivalent halves of the molecule).

Data collection: *DIF*4 (Stoe & Cie, 1992); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1992); 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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