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1,3-Di-*tert*-butyl-6-(trimethylsilylethynyl)fulvene

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In the title compound, $C_{19}H_{30}Si$, the double bonds are essentially localized. The central ring and its substituents, including the triple-bond system, are coplanar, with the Si atom lying 0.148 (2) Å out of the plane. The packing involves herring-bone layers parallel to the *ab* plane.

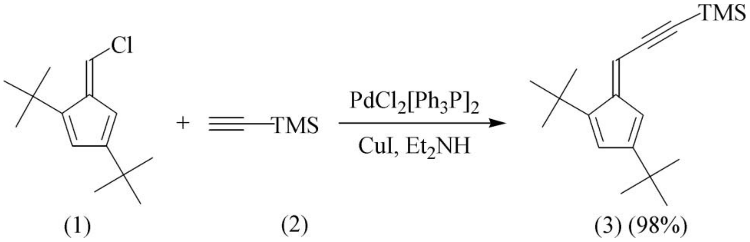
Key indicators

Single-crystal X-ray study
T = 143 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.139
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.

Comment

In our studies of the preparation and thermal behaviour of hydrocarbons containing a linearly conjugated 1,3-hexadien-5-yne subsystem, we were interested in 6-ethynylfulvene and its derivatives, because these compounds could, in principle, open up a new route to substituted pentalenes (for a summary of our work in this area, see Hopf, 2002). The title bis-*tert*-butyl derivative, (3), is one of these precursors; it can be prepared readily and in excellent yield by a Sonogashira-type coupling between the 6-chlorofulvene (1) and trimethylsilylethyne, (2). We report here the structure of (3) as determined by single-crystal X-ray diffractometry. Its spectroscopic and analytical data will be reported in the forthcoming PhD dissertation of H. Berger (Berger, 2005).



The molecule is shown in Fig. 1. Molecular dimensions (Table 1) may be regarded as normal, *e.g.* bond lengths corresponding to essentially localized multiple bonds. Atoms C1—C9 and C13 are coplanar (r.m.s. deviation 0.0173 Å), with the Si atom lying 0.148 (2) Å out of the plane thus defined.

The packing (Fig. 2) involves herring-bone layers of molecules parallel to the *ab* plane at $z \simeq \frac{1}{4}$. Within the layers, molecules are related by the 2_1 screw axis; a second layer at $z \simeq \frac{3}{4}$ is generated by inversion (and the glide plane).

We have recently published the structure of another trimethylsilylethynyl-substituted hydrocarbon (Berger *et al.*, 2004).

Experimental

Details of the synthesis of the title compound are given in Berger (2005). Single crystals were obtained by slow evaporation of an acetone solution.

Crystal data

$C_{19}H_{30}Si$
$M_r = 286.52$
Monoclinic, $P2_1/n$
$a = 9.966$ (2) Å
$b = 12.079$ (2) Å
$c = 16.345$ (2) Å
$\beta = 92.42$ (2)°
$V = 1965.8$ (6) Å^3
$Z = 4$

$D_x = 0.968$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 60 reflections
$\theta = 10$–11.5°
$\mu = 0.11$ mm^{-1}
$T = 143$ (2) K
Prism, red
0.6 × 0.3 × 0.3 mm

Data collection

Stoe Stadi-4 diffractometer
ω–θ scans
Absorption correction: none
3599 measured reflections
3395 independent reflections
2628 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$

$\theta_{\rm max} = 25.0$°
$h = -11 \rightarrow 0$
$k = -14 \rightarrow 0$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.139$
$S = 1.03$
3395 reflections
209 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 0.5585P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.15$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.345 (3)	C4—C5	1.452 (3)
C1—C5	1.488 (2)	C5—C6	1.348 (2)
C2—C3	1.474 (3)	C6—C7	1.424 (3)
C3—C4	1.342 (2)	C7—C8	1.205 (3)
C8—C7—C6	175.6 (2)	C7—C8—Si	177.9 (2)

The trimethylsilyl group is disordered over two positions with a common silicon site and relative occupancy 0.54:0.46 (2). An extensive system of restraints was used to improve stability of the refinement; the final *SHELXL* instruction file is included in the archived CIF in the supplementary material. All methyl H atoms were positioned geometrically at ideally staggered positions and refined using a riding model, with C—H = 0.98 Å and H—C—H 109.5°, and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *DIF4* (Stoe & Cie, 1992); cell refinement: *DIF4*; data reduction: *REDU4* (Stoe & Cie, 1992); 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*.

We thank Mr A. Weinkauf for technical assistance.

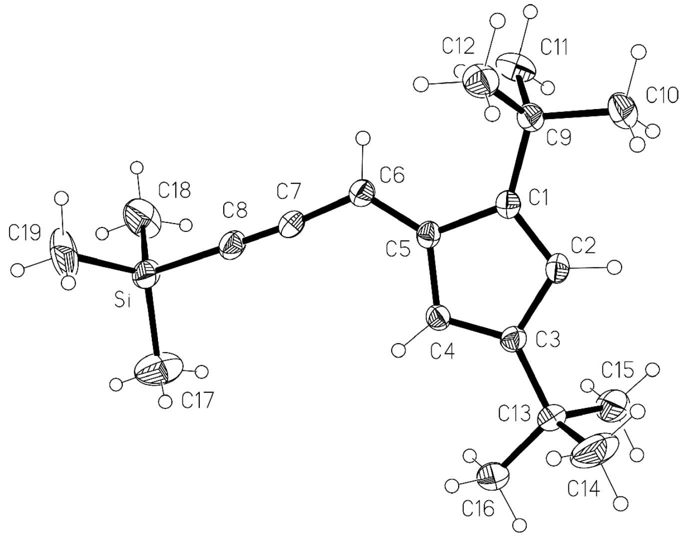


Figure 1
The molecule of the title compound. The second disorder site of the trimethylsilyl group has been omitted for clarity. Displacement ellipsoids are drawn at the 30% probability level. H atom radii are arbitrary.

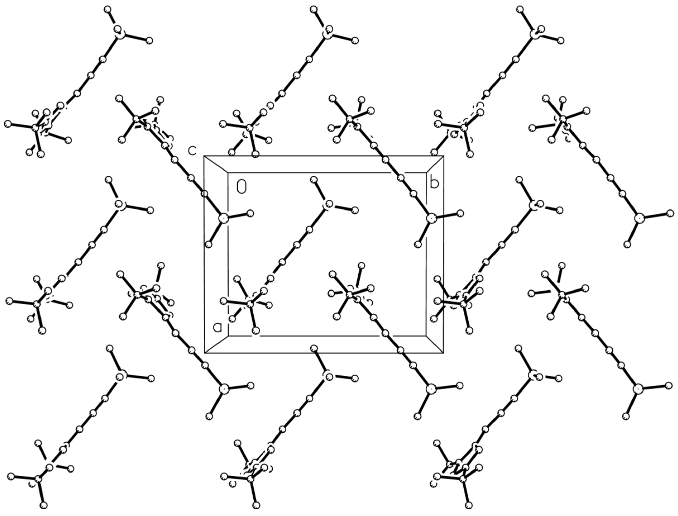


Figure 2
Packing diagram of the title compound, showing a view of the layer at $z \simeq \frac{1}{4}$. Radii are arbitrary, and the H atoms and second disorder component have been omitted.

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