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

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Key indicators

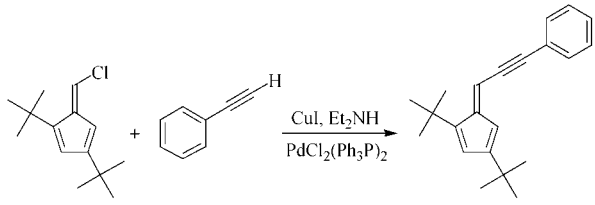
Single-crystal X-ray study
T = 143 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.141
Data-to-parameter ratio = 15.6

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: 1,3-di-*tert*-butyl-5-(3-phenylprop-2-ynylidene)cyclopenta-1,3-diene], $C_{22}H_{26}$, the rings subtend an interplanar angle of 15.7 (1)°. The triple bond length is 1.205 (3) Å.

Comment

For our studies of the chemical behaviour of new acetylenes containing a 1,3-hexadien-5-yne subsystem (Eshdat *et al.*, 2002) we required a sample of 1,3-di-*tert*-butyl-6-phenylethynyl-fulvene (3), which 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 rings subtend an interplanar angle of 15.7 (1)°.

The packing involves no unusually short contacts. The butyl groups occupy the regions at $y \simeq 0, \frac{1}{2}$, *etc* and the phenyl groups the regions at $y \simeq \frac{1}{4}, \frac{3}{4}$, *etc*.

Experimental

The title compound (3) was prepared in 94% yield by coupling the 6-chlorofulvene (1) with phenylacetylene (2) in diethylamine in the presence of CuI and $PdCl_2(Ph_3P)_2$ (Berger, 2004). Single crystals were obtained by slow cooling of solutions of (3) in acetone.

Crystal data

$C_{22}H_{26}$
M_r = 290.43
Monoclinic, $P2_1/n$
a = 6.518 (2) Å
b = 28.543 (6) Å
c = 10.194 (2) Å
β = 106.19 (3)°
V = 1821.3 (8) Å^3
Z = 4
D_x = 1.059 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 56 reflections
θ = 10–11.5°
μ = 0.06 mm^{-1}
T = 143 (2) K
Prism, red
0.70 × 0.25 × 0.25 mm

Data collection

Stoe Stadi-4 diffractometer
ω scans
Absorption correction: none
6153 measured reflections
3200 independent reflections
2436 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.0°
$h = -7 \rightarrow 0$
$k = -33 \rightarrow 21$
$l = -11 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.4622P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.053$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.141$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
$S = 1.03$	$\Delta\rho_{min} = -0.25$ e Å^{-3}
3200 reflections	
205 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C6—C7	1.410 (3)	C8—C9	1.427 (3)
C7—C8	1.205 (3)		
C6—C5—C4	124.38 (17)	C8—C7—C6	178.4 (2)
C5—C6—C7	123.90 (17)	C7—C8—C9	177.97 (19)

Methyl H atoms were located in difference syntheses, idealized (C—H 0.98 Å, H—C—H 109.5°) and refined on the basis of rigid groups allowed to rotate but not tip. Other H atoms were included at calculated positions and refined 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: *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.

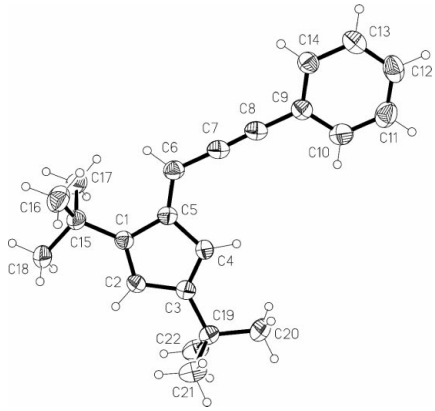


Figure 1
The molecule of the title compound in the crystal structure. Displacement ellipsoids are drawn at the 50% probability level. H-atom radii are arbitrary.

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References

Berger, H. (2004). PhD thesis. Technical University of Braunschweig, Germany.
Eshdat, L., Berger, H., Hopf, H. & Rabinovitz, M. (2002). *J. Am. Chem. Soc.* 124, 3822–3823.
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.
Stoe & Cie (1992). *DIF*4 and *REDU*4. Stoe and Cie, Darmstadt, Germany.