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3,3,6,6-Tetraphenyl-*trans*-tricyclo[3.1.0.0^{2,4}]hexane

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Key indicators

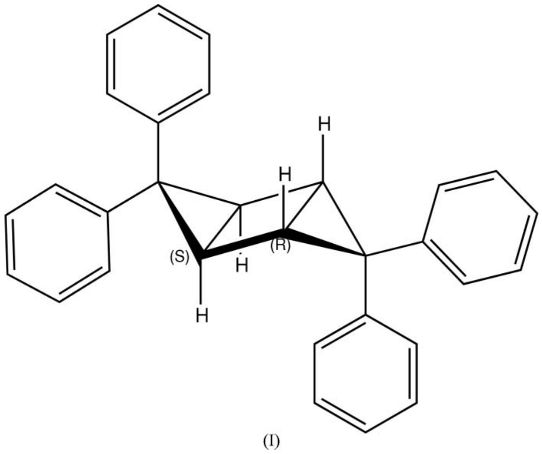
Single-crystal X-ray study
T = 85 K
Mean σ(C–C) = 0.002 Å
R factor = 0.048
wR factor = 0.120
Data-to-parameter ratio = 10.8

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

The title compound, $C_{30}H_{24}$, was produced serendipitously whilst investigating the reactions of 3,3-diphenylcyclopropane with low-valent metal substrates. There are only a small number of crystal structure determinations of products from metal-catalysed (2 + 2)-cycloadditions of cyclopropenes. The product here is the centrosymmetric *trans* isomer.

Comment

While exploring the interaction between $Os(CO)_2(PPh_3)_3$ (Collins *et al.*, 1982) and 3,3-diphenylcyclopropene (Huang *et al.*, 2003) we observed the crystallization, in very low yield and along with several other products (all osmium organometallic compounds), of the [2 + 2]-cyclodimerized product 3,3,6,6-tetraphenyl-*trans*-tricyclo[3.1.0.0^{2,4}]hexane. Cyclodimerization of cyclopropenes has previously been reported either directly by photolysis (Klimova *et al.*, 2000) or through catalysis by low oxidation state metal complexes, *e.g.* with nickel catalysts (Binger & Doyle, 1978) and with copper catalysts (Baird *et al.*, 1987). The molecule possesses a crystallographic centre of symmetry. The principal bond lengths and angles are listed in Table 1. All values are consistent with those previously obtained for other tricyclohexane structures where substitution is only on the apical C atom of the cyclopropane rings (Klimova *et al.*, 2000; Arrowood & Kass, 1999).



(I)

Experimental

Crystals of (I) were obtained as a very minor by-product of the reaction between $Os(CO)_2(PPh_3)_3$ and 3,3-diphenylcyclopropene. We have not attempted to develop reproducible reaction conditions.

Crystal data

$C_{30}H_{24}$
$M_r = 384.49$
Monoclinic, $P2_1/c$
$a = 8.1918\ (5)$ Å
$b = 18.4683\ (12)$ Å
$c = 7.0314\ (4)$ Å
$\beta = 98.709\ (1)°$
$V = 1051.51\ (11)$ Å^3
$Z = 2$

$D_x = 1.214$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4253 reflections
$\theta = 2.2–25.7°$
$\mu = 0.07$ mm^{-1}
$T = 85\ (2)$ K
Thick plate, colourless
$0.28 \times 0.22 \times 0.12$ mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.924$, $T_{\max} = 0.970$
5914 measured reflections

1994 independent reflections
1630 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 25.7°$
$h = -9 \rightarrow 9$
$k = -22 \rightarrow 22$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.120$
$S = 1.09$
1994 reflections
184 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.4478P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C4	1.504 (2)	C1—C2	1.523 (2)
C1—C3	1.514 (2)	C2—C3$^{\rm i}$	1.496 (2)
C1—C10	1.517 (2)	C2—C3	1.540 (2)
C4—C1—C3	119.10 (14)	C3$^{\rm i}$—C2—C1	112.10 (14)
C4—C1—C10	116.87 (14)	C3$^{\rm i}$—C2—C3	90.32 (13)
C3—C1—C10	115.29 (14)	C1—C2—C3	59.23 (11)
C4—C1—C2	118.95 (14)	C2$^{\rm i}$—C3—C1	111.89 (14)
C3—C1—C2	60.93 (11)	C2$^{\rm i}$—C3—C2	89.68 (13)
C10—C1—C2	113.88 (14)	C1—C3—C2	59.83 (11)

Symmetry code: (i) $-x + 1, -y, -z$.

H atoms were located in a difference Fourier map and refined with individual isotropic displacement parameters [C—H = 0.946 (19)–1.027 (19) Å].

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXTL* (Siemens, 1995).

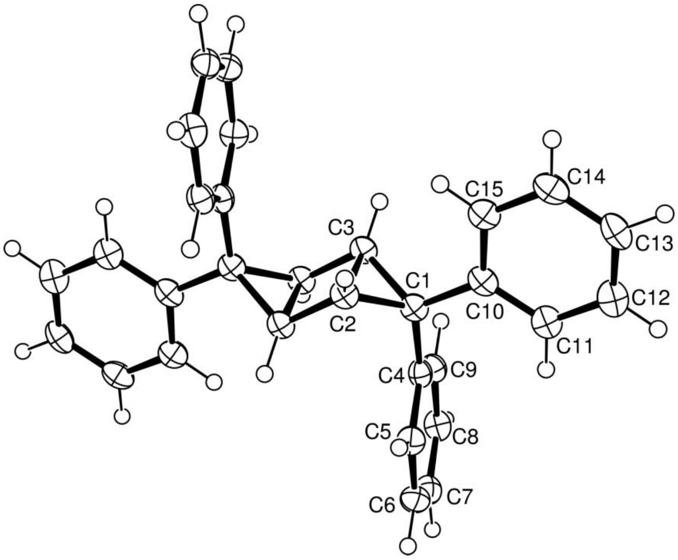


Figure 1
Structure showing 50% probability displacement ellipsoids for non-H atoms and H atoms as arbitrary spheres (Burnett & Johnson, 1996). [Symmetry code for unlabelled atoms: $1 - x, -y, -z$.]

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