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6,6-Dimethyl-2-(1-methylethenyl)-3,4,5-tris(1-methylethylidene)-1-(2-methylpropen-1-yl)cyclohexane

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Key indicators

Single-crystal X-ray study
T = 143 K
Mean σ(C–C) = 0.003 Å
R factor = 0.064
wR factor = 0.164
Data-to-parameter ratio = 16.1

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

The title compound, $C_{24}H_{36}$, crystallizes with two independent but very similar molecules in the asymmetric unit. The ring torsion angles about the double bonds are *ca* 13°; the ring sp^2 bond angles at the three radialene-type junctions are *ca* 110°.

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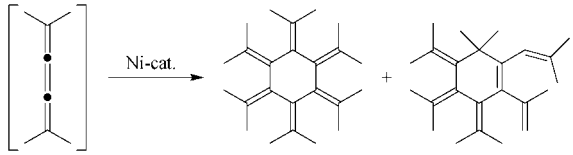
Comment

Permethylbutatriene, (1), generated *in situ*, is known to cyclooligomerize to dodecamethyl[6]radialene, (2), in the presence of nickel catalyst systems (Iyoda *et al.*, 1984, 1988; Stehling & Wilke, 1985). Although the yield of this process is good (>70%), it is accompanied by the formation of several by-products, most of which have not been fully characterized. Repeating this trimerization (Höpfner, 1996), we have isolated one of the minor (5%) products and have identified it, by X-ray methods, as the title compound, (3).



(1) (2) (3)

Compound (3) crystallizes with two independent but very similar molecules in the asymmetric unit (Fig. 1); a least-squares fit of all non-H atoms (Fig. 2) gives an r.m.s. deviation of only 0.04 Å. For this reason, only the dimensions of molecule 1 (unprimed atoms) will be discussed explicitly.

The ring shows a torsion angle of 13.8 (3)° about the C2═C3 double bond. The sp^2 angles at the radialene-type ring junctions are markedly narrowed, with ring angles of 109.2 (2), 109.9 (2) and 112.9 (2)° at C4, C5 and C6, respectively. Other molecular dimensions may be regarded as normal (*e.g.* the double-bond lengths in Table 1), but taking note of the caveat given in the *Experimental* section.

Experimental

Compound (3) was obtained as described by Höpfner (1996) and recrystallized from methanol.

Crystal data

$C_{24}H_{36}$
M_r = 324.53
Monoclinic, $P2_1/n$
a = 14.382 (2) Å
b = 17.062 (2) Å
c = 17.053 (2) Å
β = 95.436 (12)°
V = 4165.6 (9) Å^3
Z = 8
D_x = 1.035 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 54 reflections
θ = 10–11°
μ = 0.06 mm^{-1}
T = 143 (2) K
Prism, colourless
0.6 × 0.5 × 0.4 mm

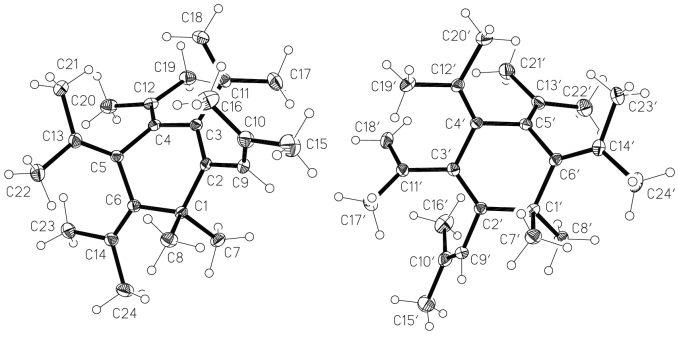


Figure 1
The two independent molecules of compound (3) in the crystal. Displacement ellipsoids are drawn at the 30% probability level and H-atom radii are arbitrary.

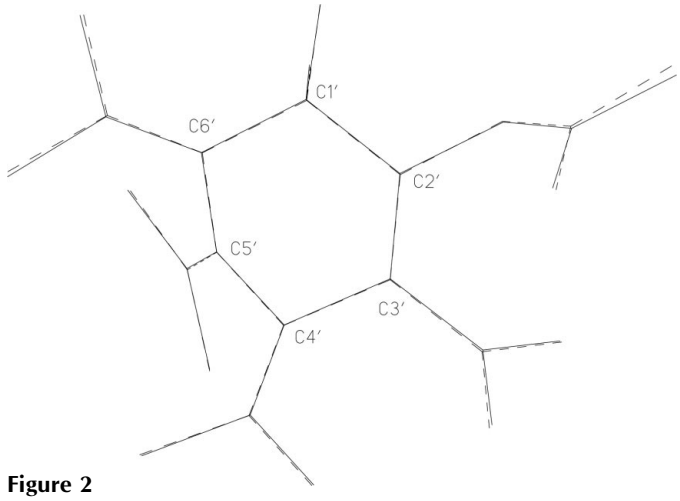


Figure 2
Least-squares fit of both independent molecules of compound (3).

Data collection

Stoe Stadi-4 diffractometer	$\theta_{max} = 25.0°$
ω–θ scans	$h = -17 \rightarrow 17$
Absorption correction: none	$k = 0 \rightarrow 20$
7622 measured reflections	$l = -20 \rightarrow 1$
7330 independent reflections	3 standard reflections
4782 reflections with $I > 2\sigma(I)$	frequency: 60 min
$R_{int} = 0.025$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 2.454P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.064$	$(\Delta/\sigma)_{max} = 0.036$
$wR(F^2) = 0.165$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.27$ e Å^{-3}
7330 reflections	
455 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C2—C3	1.350 (3)	C2′—C3′	1.354 (3)
C4—C12	1.344 (3)	C4′—C12′	1.340 (3)
C5—C13	1.340 (3)	C5′—C13′	1.334 (3)
C6—C14	1.339 (3)	C6′—C14′	1.338 (3)
C9—C10	1.321 (4)	C9′—C10′	1.328 (4)
C11—C18	1.367 (4)	C11′—C18′	1.362 (4)
C2—C1—C6	112.08 (19)	C2′—C1′—C6′	112.14 (19)
C3—C2—C1	122.7 (2)	C3′—C2′—C1′	122.9 (2)
C2—C3—C4	118.6 (2)	C2′—C3′—C4′	118.7 (2)
C3—C4—C5	109.21 (19)	C3′—C4′—C5′	109.3 (2)
C6—C5—C4	109.9 (2)	C6′—C5′—C4′	110.3 (2)
C5—C6—C1	112.90 (19)	C5′—C6′—C1′	112.57 (19)
C6—C1—C2—C3	−22.3 (3)	C6′—C1′—C2′—C3′	−21.3 (3)
C1—C2—C3—C4	13.8 (3)	C1′—C2′—C3′—C4′	13.3 (3)
C2—C3—C4—C5	29.5 (3)	C2′—C3′—C4′—C5′	29.2 (3)
C3—C4—C5—C6	−64.5 (2)	C3′—C4′—C5′—C6′	−64.2 (2)
C4—C5—C6—C1	55.9 (3)	C4′—C5′—C6′—C1′	56.0 (3)
C2—C1—C6—C5	−13.6 (3)	C2′—C1′—C6′—C5′	−14.2 (3)

Methyl H atoms were identified in difference syntheses, idealized and then refined using rigid methyl groups (C—H = 0.98 Å and H—C—H = 109.5°) Other H atoms were included using a riding model with fixed C—H bond lengths of 0.95 Å; U_{iso}(H) values were fixed at 1.2 times the U_{eq} values of the parent atom. The bond lengths of the methylethenyl substituent, and the fact that the largest difference peaks were at bonding distance to C17 and C17′ (the corresponding methyl H atoms were the least well resolved) may indicate some disorder between the methylene and methyl sites C17 and C18 (and C17′ and C18′).

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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