C10—C1—C2—C11	179.3 (2)	C11—C12—C13—C14	176.3 (2)
O20—C11—C12—C13	−20.8 (4)	C12—C13—C14—C19	170.8 (2)
C2—C11—C12—C13	160.2 (2)		

Data collection: *SDP* (Frenz, 1978). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1049). Services for accessing these data are described at the back of the journal.

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3,4-Diiodo-2,5-dimethylhexa-2,4-diene

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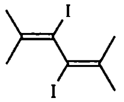
Abstract

The title compound, $C_8H_{12}I_2$, displays crystallographic twofold symmetry. The two double bonds are not conjugated [torsion angle −78.8 (5)° about the central single bond]. Intermolecular I···I contacts of 4.098 (1) Å connect the molecules into layers parallel to the *xy* plane.

Comment

As part of our general investigations on structural and spectroscopic features of π-systems (Hopf, Hänel & Traetteberg, 1994; Hopf & Hänel, 1994), we have recently become interested in orthogonal butadienes (*i.e.* those in which the planes of the π-bond systems are orthogonal). An example is provided by 2,3-di-*tert*-butyl-1,3-butadiene, for which the orthogonal structure has been established by electron diffraction (Hopf, Traetteberg & Hänel, 1994) and X-ray structure analysis (at 110 K, torsion angle 84°; Roth, Adamczak, Breuckmann, Lennartz & Boese, 1991).

The title compound, (I) (Fig. 1), displays crystallographic twofold symmetry. It can be seen that even the iodine substituents, which are only moderately bulky compared with *tert*-butyl groups, can force the two double bonds almost completely out of conjugation; the dihedral angle C2—C3—C3^{i}—C2^{i} is −78.8 (5)° [symmetry code: (i) $1-x, y, \frac{1}{2}-z$]. The central C3—C3^{i} bond is, at 1.469 (5) Å, closer to the standard value for non-conjugated C_{sp^2}—C_{sp^2} (1.478 *cf.* 1.455 Å in conjugated systems; Allen *et al.*, 1987) and C2—C3 is, at 1.334 (4) Å, close to the standard value of 1.331 Å for $R_2C{=}CR_2$.



(I)

Several bond angles display marked deviations from ideal values: C1—C2—C4 113.8 (3), C1—C2—C3 125.6 (3) and C3^{i}—C3—I 112.74 (15)°. This presumably reflects steric pressure, although it is surprising that the angle between the methyl groups is so narrow.

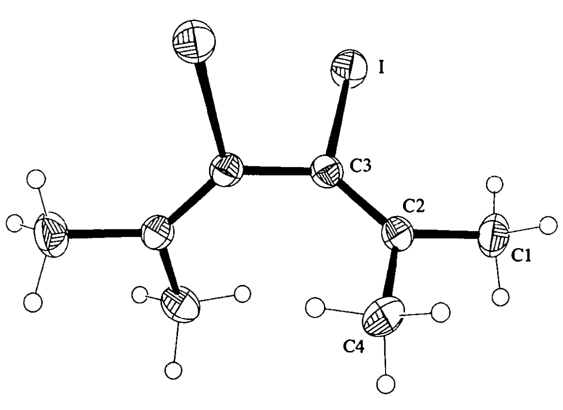


Fig. 1. The molecule of the title compound in the crystal. Ellipsoids represent 50% probability levels. Only the asymmetric unit is numbered. H-atom radii are arbitrary.

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The intramolecular I· · ·I contact is 3.954 (1) Å. Short intermolecular contacts, I· · ·I^{ii} and I· · ·I^{iii} 4.098 (1) Å, and I· · ·I· · ·I 107.45 (3)°, connect the molecules in layers parallel to the *xy* plane (Fig. 2) [symmetry codes: (ii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $\frac{1}{2}-x, y+\frac{1}{2}, \frac{1}{2}-z$].

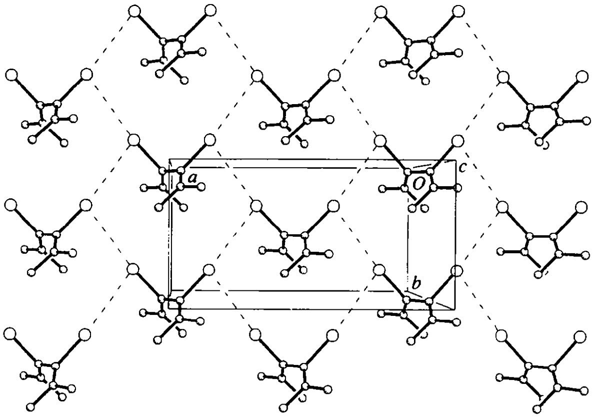

Fig. 2. Packing diagram viewed (approximately) along the *z* axis (H atoms omitted). I· · ·I contacts are indicated by dashed lines.

Experimental

The title compound was obtained by a known procedure (Zalkind, Rubin & Kruglow, 1926). Spectroscopic data: ^{1}H NMR (CDCl$_3$, Bruker AM 400): δ = 1.94 (*s*, 6H, CH$_3$), 1.71 (*s*, 6H, CH$_3$); ^{13}C NMR (CDCl$_3$, Bruker AM 400): δ = 140.14 (*s*), 100.78 (*s*), 29.83 (*q*), 20.29 (*q*); IR (KBr, Nicolet FT-IR 320): ν = 2988 (*m*), 2932 (*m*), 2906 (*s*), 2842 (*m*), 1638 (*w*), 1598 (*w*), 1438 (*m*), 1434 (*m*), 1425 (*m*), 1422 (*m*), 1401 (*w*), 1364 (*s*), 1220 (*w*), 1209 (*m*), 1065 (*m*), 999 (*s*), 846 (*m*), 825 (*s*), 705 (*s*), 670 (*w*) cm^{-1}; UV/visible (CH$_2$Cl$_2$, HP 8452 A Diode Array Spectrophotometer): λ_{max} (log ε) = 246 nm (3.972). Single crystals were obtained by sublimation.

Crystal data

$C_8H_{12}I_2$
M_r = 361.98
Monoclinic
$C2/c$
a = 12.614 (4) Å
b = 6.607 (2) Å
c = 12.921 (4) Å
β = 94.59 (3)°
V = 1073.4 (6) Å^3
Z = 4
D_x = 2.240 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 52 reflections
θ = 10.0–11.5°
μ = 5.798 mm^{-1}
T = 143 (2) K
Prism
0.60 × 0.30 × 0.25 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: ΔF (*SHELXA*; Sheldrick, unpublished)
T_{min} = 0.068, T_{max} = 0.235
2190 measured reflections
1241 independent reflections
1189 reflections with $I > 2\sigma(I)$

R_{int} = 0.0306
θ_{max} = 27.54°
$h = -16 \rightarrow 16$
$k = -8 \rightarrow 5$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0215
$wR(F^2)$ = 0.0552
S = 1.109
1241 reflections
49 parameters
H atoms: rigid methyls
$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 2.2629P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.96 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.0039 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
I	0.345810 (14)	0.30804 (3)	0.267881 (15)	0.03086 (11)
C1	0.3823 (3)	0.6706 (5)	0.4503 (2)	0.0320 (7)
C2	0.4626 (2)	0.6669 (4)	0.3708 (2)	0.0241 (5)
C3	0.4619 (2)	0.5406 (4)	0.29015 (19)	0.0218 (5)
C4	0.5494 (3)	0.8231 (5)	0.3889 (3)	0.0325 (7)

Table 2. *Selected geometric parameters (Å, °)*

I—C3	2.127 (3)	C2—C4	1.509 (4)
C1—C2	1.500 (4)	C3—C3^i	1.469 (5)
C2—C3	1.334 (4)		
C3—C2—C1	125.6 (3)	C2—C3—C3^i	126.0 (2)
C3—C2—C4	120.5 (3)	C2—C3—I	121.3 (2)
C1—C2—C4	113.8 (3)	C3^i—C3—I	112.74 (15)
C1—C2—C3—C3^i	177.7 (3)	C4—C2—C3—I	176.4 (2)
C4—C2—C3—C3^i	−2.6 (4)	C2—C3—C3^i—C2^i	−78.8 (5)
C1—C2—C3—I	−3.4 (4)	I—C3—C3^i—I^i	−76.8 (3)

Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.

The number of least-squares parameters was increased by 18 to allow for the 'hidden' parameters introduced by the ΔF absorption correction.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1291). Services for accessing these data are described at the back of the journal.

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All-*E*-7,8,9,10,11,12-hexaethyl[6]radialene†

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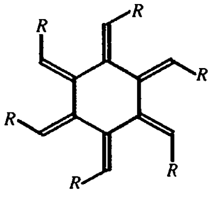
Abstract

The title compound, $C_{24}H_{36}$, displays crystallographic inversion symmetry, but the actual symmetry is close to $\bar{3}$. The structure is closely similar to, but not isostructural with, the analogous hexamethyl derivative [Marsh & Dunitz (1975). *Helv. Chim. Acta*, **58**, 707–712]. The ring adopts a chair conformation with absolute torsion angles in the range 45.4 (4)–45.8 (4)°. Ring bond lengths are 1.480 (4)–1.500 (4) Å and ring bond angles are 113.9 (3)–114.8 (3)°.

Comment

As part of our current investigations on cross-conjugated π-systems, we were interested in the structure of all-*E*-7,8,9,10,11,12-hexaethyl[6]radialene, (2) (for a review of radialenes see Hopf & Maas, 1992). X-ray structural investigations of the conformation of the cyclohexane ring in [6]radialenes have revealed both planar and non-planar geometries; annelated systems are generally planar, whereas unbridged systems are generally planar when the substituents are solely diazo and keto, and otherwise non-planar (Jones, Ahrens, Höpfner & Hopf, 1997, and references therein). The X-ray structure of the hexamethyl derivative, (1), was determined by Marsh & Dunitz (1975), revealing a molecule with crystallographic inversion symmetry (effective symmetry $\bar{3}$) and a chair conformation (torsion angles alternating ±46.2°). A recent gas-phase electron-diffraction study (Benet-Buchholz, Boese, Hauman & Traetteberg, 1997) found the bond lengths and angles to be in good agreement with those of Marsh & Dunitz (1975); however, the torsion angles in the gas phase were appreciably greater at 53.0°. To obtain further information about such systems, we have now investigated the solid-state structure of (2), the next higher homologue of (1).



(1) R = Me
(2) R = Et

Despite the lower measurement temperature the structure of (2) is slightly less precise than that of (1); this can be attributed to the greater displace-

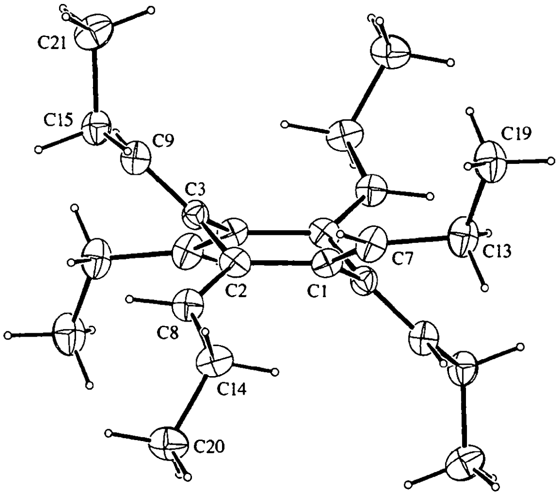


Fig. 1. The molecule of the title compound in the crystal. Ellipsoids represent 30% probability levels. Only the asymmetric unit is numbered. H-atom radii are arbitrary.

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† Alternative name: (1*E*,2*E*,3*E*,4*E*,5*E*,6*E*)-1,2,3,4,5,6-hexakis(propylidene)cyclohexane.