

O(1)—C(12)—C(11)—C(14)	−10.8 (3)
O(1)—C(13)—C(14)—C(11)	2.5 (3)
C(11)—C(12)—O(1)—C(13)	12.6 (2)
C(12)—O(1)—C(13)—C(14)	−9.8 (2)
C(12)—C(11)—C(14)—C(13)	5.4 (3)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR92* (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1189). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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C₂₀H₁₀F₈, a Divinyl Acetylene

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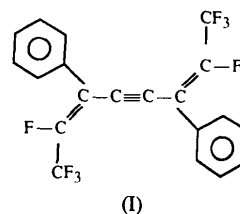
Abstract

The crystal structure of the title compound shows it to be (*E,E*)-1,1,1,2,7,8,8,8-octafluoro-3,6-diphenylocta-2,6-dien-4-yne.

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Comment

Defluorination of the *E* or *Z* isomer of 3,6-diphenyl-decafluoroocta-3,4,5-triene yielded a substituted divinyl acetylene (I) in more than 95% isomeric purity (Morken, Burton & Swenson, 1994).



The molecules pack in the crystal in stacks along the *c* axis and these stacks pack in a herring-bone fashion. The trifluoromethyl groups have high thermal motion and are rotationally disordered, each methyl group having two sets of F-atom positions. The occupancy factors for the two sets of F atoms bonded to C1 (F1A, F1B, F1C and F1D, F1E, F1F) were set to 0.5. For the other disordered CF₃ group (C8), occupancy of 0.85 for F8A, F8B, F8C and 0.15 for F8D, F8E, F8F gave similar thermal parameters for the two sets and minimal residual density in the difference map.

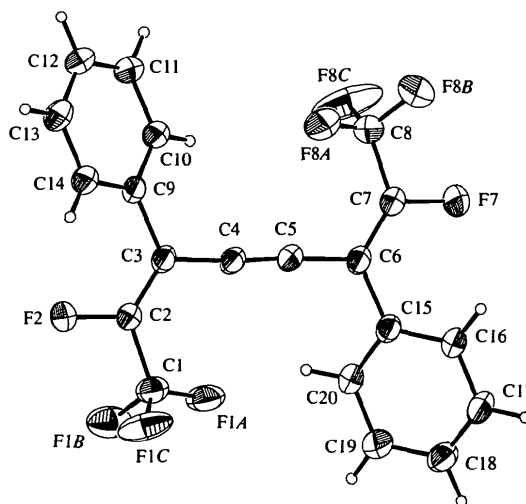


Fig. 1. View and numbering scheme of the title compound. Ellipsoids are drawn at the 35% level. Only one orientation of each disordered trifluoromethyl group is shown for clarity. H atoms are given an arbitrarily small radius.

The octadienyne portion of the molecule is essentially flat. The average deviation from the least-squares plane (C1–C9, C15, F2, F7) is 0.031 (8) Å. Steric interaction between the *ortho* position atoms in the phenyl rings and the vinyl fluoride atoms prevents the phenyl rings from being coplanar with the dienyne plane. The dihedral angles between the dienyne plane (C1–C9, C15, F2, F7) and the phenyl ring planes are 34.4 (1)° (for ring C9–C14) and 35.3 (1)° (for ring C15–C20).

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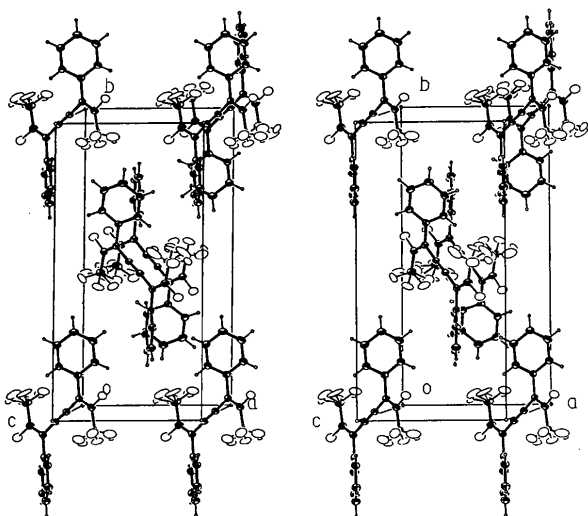


Fig. 2. Stereoview showing packing in the unit cell. Both orientations of the two disordered CF₃ groups are shown. Ellipsoids are drawn at the 20% level. H atoms are given an arbitrarily small displacement parameter.

Experimental

The title compound was prepared as described by Morken *et al.* (1994) and recrystallized from hexane.

Crystal data

C₂₀H₁₀F₈

$M_r = 402.29$

Monoclinic

$P2_1/n$

$a = 9.748 (2) \text{ \AA}$

$b = 19.080 (3) \text{ \AA}$

$c = 9.955 (2) \text{ \AA}$

$\beta = 109.14 (1)^\circ$

$V = 1749 (1) \text{ \AA}^3$

$Z = 4$

$D_x = 1.53 \text{ Mg m}^{-3}$

D_m not measured

Data collection

Enraf–Nonius CAD-4 diffractometer

$\theta/2\theta$ scans

Absorption correction:

empirical, from 3 ψ scans at 10° intervals (MolEN; Fair, 1990)

$T_{\min} = 0.9303$, $T_{\max} = 0.9985$

7137 measured reflections

2690 independent reflections

Refinement

Refinement on F

$R = 0.050$

$wR = 0.076$

Cu $K\alpha$ radiation

$\lambda = 1.54184 \text{ \AA}$

Cell parameters from 22 reflections

$\theta = 19\text{--}39^\circ$

$\mu = 1.307 \text{ mm}^{-1}$

$T = 291 \text{ K}$

Needle, c axis along needle direction

$0.45 \times 0.15 \times 0.10 \text{ mm}$

Colorless

2114 observed reflections [$I > 2.0\sigma(I)$]

$R_{\text{int}} = 0.039$

$\theta_{\text{max}} = 70.0^\circ$

$h = -11 \rightarrow 11$

$k = -23 \rightarrow 23$

$l = -12 \rightarrow 0$

4 standard reflections

frequency: 240 min

intensity decay: 2.01%

Weighting scheme based

on measured e.s.d.'s

$(\Delta/\sigma)_{\text{max}} = 0.077$

$S = 1.46$
2114 reflections
307 parameters
H atoms riding, $U = 1.3 \times U(\text{bonded atom})$

$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

	x	y	z	U_{eq}
C1	0.2955 (3)	-0.0305 (1)	0.0422 (3)	0.141 (2)
C2	0.2610 (2)	0.0440 (1)	-0.0082 (3)	0.111 (1)
C3	0.1555 (2)	0.06761 (9)	-0.1213 (2)	0.099 (1)
C4	0.0531 (2)	0.01972 (9)	-0.2116 (2)	0.103 (1)
C5	-0.0374 (2)	-0.01702 (9)	-0.2910 (2)	0.104 (1)
C6	-0.1403 (2)	-0.06484 (9)	-0.3807 (2)	0.106 (1)
C7	-0.2484 (3)	-0.0409 (1)	-0.4908 (3)	0.130 (2)
C8	-0.2824 (3)	0.0340 (1)	-0.5410 (3)	0.156 (2)
C9	0.1349 (2)	0.14358 (9)	-0.1607 (2)	0.097 (1)
C10	-0.0039 (2)	0.1701 (1)	-0.2206 (3)	0.112 (1)
C11	-0.0256 (3)	0.2405 (1)	-0.2569 (3)	0.130 (2)
C12	0.0931 (3)	0.2844 (1)	-0.2341 (3)	0.139 (2)
C13	0.2309 (3)	0.2583 (1)	-0.1759 (3)	0.142 (2)
C14	0.2533 (3)	0.1881 (1)	-0.1390 (3)	0.119 (1)
C15	-0.1227 (2)	-0.14073 (9)	-0.3400 (2)	0.100 (1)
C16	-0.1549 (3)	-0.1932 (1)	-0.4439 (3)	0.123 (1)
C17	-0.1387 (3)	-0.2631 (1)	-0.4034 (3)	0.144 (2)
C18	-0.0906 (3)	-0.2809 (1)	-0.2623 (3)	0.133 (2)
C19	-0.0567 (3)	-0.2296 (1)	-0.1590 (3)	0.134 (2)
C20	-0.0721 (3)	-0.1594 (1)	-0.1996 (3)	0.117 (1)
F1A	0.1984 (4)	-0.0746 (1)	-0.0045 (5)	0.263 (3)
F1B	0.3083 (5)	-0.0326 (2)	0.1862 (4)	0.215 (3)
F1C	0.4199 (3)	-0.0497 (2)	0.0454 (5)	0.235 (2)
F1D	0.1931 (4)	-0.0587 (2)	0.0750 (4)	0.258 (3)
F1E	0.4158 (5)	-0.0384 (2)	0.1300 (6)	0.344 (3)
F1F	0.2880 (4)	-0.0724 (1)	-0.0738 (4)	0.200 (3)
F2	0.3524 (2)	0.08790 (6)	0.0843 (2)	0.153 (1)
F7	-0.3513 (2)	-0.08365 (7)	-0.5725 (2)	0.192 (1)
F8A	-0.1664 (3)	0.07264 (9)	-0.5154 (3)	0.227 (2)
F8B	-0.3367 (3)	0.0360 (1)	-0.6802 (3)	0.242 (2)
F8C	-0.3649 (3)	0.0641 (1)	-0.4865 (3)	0.39 (2)
F8D	-0.421 (1)	0.0400 (4)	-0.616 (1)	0.175 (2)
F8E	-0.279 (1)	0.0714 (4)	-0.430 (1)	0.286 (8)
F8F	-0.188 (1)	0.0654 (6)	-0.578 (1)	0.48 (1)

Table 2. Geometric parameters (\AA , $^\circ$)

C1—C2	1.508 (3)	C8—F8B	1.312 (4)
C1—F1A	1.237 (6)	C8—F8C	1.248 (5)
C1—F1B	1.398 (6)	C8—F8D	1.32 (1)
C1—F1C	1.257 (6)	C8—F8E	1.31 (2)
C1—F1D	1.267 (7)	C8—F8F	1.25 (3)
C1—F1E	1.220 (7)	C9—C10	1.383 (3)
C1—F1F	1.386 (5)	C9—C14	1.392 (3)
C2—C3	1.329 (3)	C10—C11	1.389 (3)
C2—F2	1.344 (3)	C11—C12	1.385 (4)
C3—C4	1.432 (3)	C12—C13	1.370 (4)
C3—C9	1.498 (3)	C13—C14	1.387 (3)
C4—C5	1.199 (3)	C15—C16	1.399 (3)
C5—C6	1.431 (3)	C15—C20	1.367 (4)
C6—C7	1.328 (4)	C16—C17	1.387 (3)
C6—C15	1.498 (3)	C17—C18	1.370 (4)
C7—C8	1.513 (4)	C18—C19	1.379 (4)
C7—F7	1.342 (3)	C19—C20	1.393 (3)
C8—F8A	1.303 (5)		
C2—C1—F1A	117.1 (3)	C7—C8—F8B	110.2 (3)
C2—C1—F1B	107.6 (3)	C7—C8—F8C	113.2 (3)
C2—C1—F1C	113.1 (4)	C7—C8—F8D	110.0 (7)
C2—C1—F1D	112.1 (4)	C7—C8—F8E	107 (1)
C2—C1—F1E	114.3 (4)	C7—C8—F8F	116. (1)
C2—C1—F1F	108.7 (3)	F8A—C8—F8B	102.3 (4)

F1A—C1—F1B	99.5 (6)	F8A—C8—F8C	107.2 (4)
F1A—C1—F1C	115.2 (6)	F8B—C8—F8C	110.6 (5)
F1B—C1—F1C	101.8 (5)	F8D—C8—F8E	99. (1)
F1D—C1—F1E	114.6 (7)	F8D—C8—F8F	121. (2)
F1D—C1—F1F	98.0 (5)	F8E—C8—F8F	100. (2)
F1E—C1—F1F	107.6 (7)	C3—C9—C10	119.7 (2)
C1—C2—C3	128.9 (2)	C3—C9—C14	121.1 (2)
C1—C2—F2	109.5 (2)	C10—C9—C14	119.2 (2)
C3—C2—F2	121.6 (2)	C9—C10—C11	120.6 (2)
C2—C3—C4	120.1 (2)	C10—C11—C12	119.6 (2)
C2—C3—C9	123.4 (2)	C11—C12—C13	120.0 (2)
C4—C3—C9	116.5 (2)	C12—C13—C14	120.6 (2)
C3—C4—C5	176.1 (2)	C9—C14—C13	119.9 (2)
C4—C5—C6	176.2 (2)	C6—C15—C16	120.9 (2)
C5—C6—C7	120.0 (2)	C6—C15—C20	120.0 (2)
C5—C6—C15	116.9 (2)	C16—C15—C20	119.2 (2)
C7—C6—C15	123.1 (2)	C15—C16—C17	119.7 (3)
C6—C7—C8	128.8 (2)	C16—C17—C18	120.4 (2)
C6—C7—F7	121.6 (2)	C17—C18—C19	120.4 (2)
C8—C7—F7	109.6 (2)	C18—C19—C20	119.3 (3)
C7—C8—F8A	112.8 (3)	C15—C20—C19	121.1 (2)
C1—C2—C3—C4	2.0 (4)	C4—C5—C6—C7	-177 (4)
C1—C2—C3—C9	-178.9 (2)	C4—C5—C6—C15	6 (4)
F2—C2—C3—C4	-175.2 (2)	C5—C6—C7—C8	0.5 (4)
F2—C2—C3—C9	4.0 (4)	C5—C6—C7—F7	-176.3 (2)
C2—C3—C4—C5	169 (4)	C15—C6—C7—C8	177.7 (3)
C9—C3—C4—C5	-10 (4)	C15—C6—C7—F7	0.9 (4)
C2—C3—C9—C10	-144.7 (2)	C5—C6—C15—C16	-144.9 (2)
C2—C3—C9—C14	35.8 (3)	C5—C6—C15—C20	33.9 (3)
C4—C3—C9—C10	34.5 (3)	C7—C6—C15—C16	37.8 (4)
C4—C3—C9—C14	-145.0 (2)	C7—C6—C15—C20	-143.4 (3)
C3—C4—C5—C6	-175 (3)		

Backgrounds were obtained from analysis of the scan profile (Blessing, Coppens & Becker, 1974)

Data collection: *CAD-4* (Enraf–Nonius, 1977). Cell refinement: *CAD-4*. Data reduction: *PROCESS MolEN* (Fair, 1990). Program(s) used to solve structure: direct methods (*MULTAN*; Main *et al.*, 1980). Program(s) used to refine structure: *LSFM MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *CIF VAX (MolEN)*.

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Lists of structure factors, least-squares-planes data, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1151). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,3,5-Tri-*tert*-butyl-5-(1,3,5-tri-*tert*-butyl-4-oxo-2,5-cyclohexadienyl)methylbicyclo-[4.1.0]hept-3-en-2-one, a Highly Hindered Propane

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Abstract

The title compound, C₃₈H₆₂O₂, is one of the reaction products of diazomethane with two equivalents of 2,4,6-tri-*tert*-butylphenoxy. In the crystal structure, the cyclopropane ring is almost perpendicular to the basal plane of the boat-configured cyclohexenone ring.

Comment

2,4,6-Tri-*tert*-butylphenoxy, (1), reacts with diazomethane to give two formal 1:1 addition products, (2) and (3), after elimination of N₂ (Müller, Renner & Rieker, 1968; Rieker, Renner & Müller, 1969). The structures of (2a) and (3) were originally derived from chemical reactivity and ¹H NMR (60 MHz) also using deuteriated analogues of (2) and (3) obtained from CD₂N₂ or 3,5-dideuterio-2,4,6-tri-*tert*-butylphenoxy.

