

## Refinement

Refinement on  $F$   
 $R = 0.054$   
 $wR = 0.056$   
 $S = 1.58$   
 808 reflections  
 109 parameters  
 H atoms not refined  
 $w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.36 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.32 \text{ e } \text{Å}^{-3}$   
 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 ( $\text{Å}^2$ )

$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	x	y	z	$B_{\text{eq}}$
Cl(1)	0.7493 (3)	-0.1490 (1)	0.6718 (1)	3.18 (6)
O(1)	0.4008 (9)	-0.0280 (2)	0.8031 (4)	3.6 (2)
O(2)	0.2749 (8)	0.0190 (2)	0.5998 (4)	3.1 (2)
O(3)	-0.494 (1)	0.2209 (2)	0.6696 (4)	3.6 (2)
N(1)	-0.175 (1)	0.1182 (3)	0.5533 (4)	2.7 (2)
C(1)	0.067 (1)	0.0759 (3)	0.7776 (5)	2.2 (2)
C(2)	-0.129 (1)	0.1237 (3)	0.6994 (5)	2.3 (2)
C(3)	-0.302 (1)	0.1778 (3)	0.7552 (6)	2.6 (3)
C(4)	-0.274 (1)	0.1842 (4)	0.8927 (6)	3.1 (3)
C(5)	-0.080 (1)	0.1364 (4)	0.9714 (5)	3.2 (3)
C(6)	0.089 (1)	0.0834 (4)	0.9159 (6)	3.0 (3)
C(7)	0.252 (1)	0.0198 (4)	0.7179 (6)	2.5 (3)

Table 2. Selected geometric parameters ( $\text{Å}$ ,  $^\circ$ )

O(1)—C(7)	1.320 (6)	C(1)—C(7)	1.469 (8)
O(2)—C(7)	1.221 (6)	C(2)—C(3)	1.394 (8)
O(3)—C(3)	1.366 (7)	C(3)—C(4)	1.394 (8)
N(1)—C(2)	1.477 (6)	C(4)—C(5)	1.382 (8)
C(1)—C(2)	1.386 (7)	C(5)—C(6)	1.370 (8)
C(1)—C(6)	1.405 (7)		
C(2)—C(1)—C(6)	118.1 (5)	C(2)—C(3)—C(4)	119.0 (6)
C(2)—C(1)—C(7)	121.1 (5)	C(3)—C(4)—C(5)	119.8 (6)
C(6)—C(1)—C(7)	120.8 (5)	C(4)—C(5)—C(6)	120.8 (5)
N(1)—C(2)—C(1)	122.0 (5)	C(1)—C(6)—C(5)	120.7 (5)
N(1)—C(2)—C(3)	116.4 (5)	O(1)—C(7)—O(2)	122.5 (6)
C(1)—C(2)—C(3)	121.5 (5)	O(1)—C(7)—C(1)	114.6 (5)
O(3)—C(3)—C(2)	117.0 (5)	O(2)—C(7)—C(1)	122.9 (6)
O(3)—C(3)—C(4)	124.0 (6)		

H atoms were located from difference Fourier maps, except for atoms H(22) and H(23), which were placed in calculated positions. All of these H atoms were included in subsequent least-squares refinement calculations with fixed isotropic displacement parameters.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELX86* (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: TA1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 9,10,12,14-Tetrahydro-12,12,14,14-tetra-phenyl-9,10[3',4']-furanoanthracene

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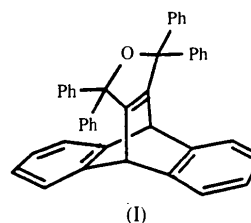
(Received 4 April 1996; accepted 14 May 1996)

## Abstract

The title molecule,  $\text{C}_{42}\text{H}_{30}\text{O}$ , is the product of solid-state photolysis of inclusion complexes of a dibenzobarrelene-bis(diphenylmethanol), and has normal geometry and dimensions.

## Comment

The title compound, (I), was studied to assist in the interpretation of the photolysis of inclusion complexes of 9,10-dihydro-9,10-ethenoanthracene-11,12-bis(diphenyl-



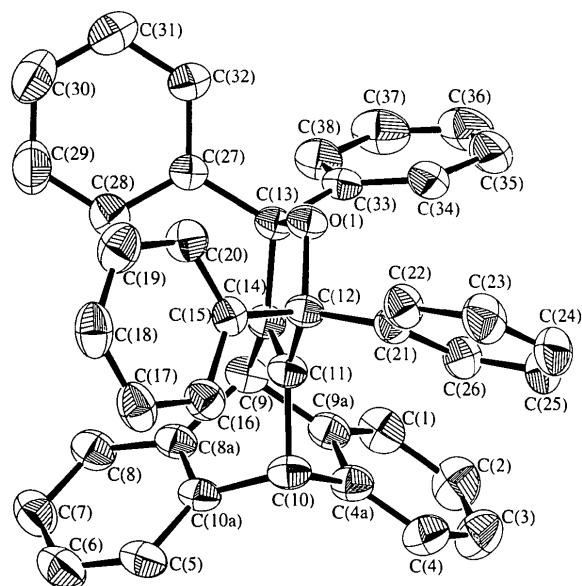


Fig. 1. View of the molecule with 33% probability displacement ellipsoids and the crystallographic numbering system.

methanol) (Fu, Olovsson, Scheffer & Trotter, 1995); the photolysis reaction results in elimination of water, and formation of a furan ring (Fig. 1).

## Experimental

The compound was obtained by photolysis of crystalline inclusion complexes of 9,10-dihydro-9,10-ethenoanthracene-11,12-bis(diphenylmethanol) (Fu, Olovsson, Scheffer & Trotter, 1995).

### Crystal data

C<sub>42</sub>H<sub>30</sub>O

*M<sub>r</sub>* = 550.70

Monoclinic

*P*2<sub>1</sub>/*c*

*a* = 9.969 (2) Å

*b* = 15.883 (1) Å

*c* = 19.369 (2) Å

β = 95.20 (1)°

*V* = 3054.2 (6) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.198 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

Cu *K*α radiation

λ = 1.5418 Å

Cell parameters from 25 reflections

θ = 22.8–27.9°

μ = 0.54 mm<sup>-1</sup>

*T* = 294 K

Prism

0.30 × 0.19 × 0.09 mm

Colorless

### Data collection

Rigaku AFC-6S diffractometer

ω–2θ scans

Absorption correction:

ψ scans (North, Phillips & Matthews, 1968)

*T<sub>min</sub>* = 0.947, *T<sub>max</sub>* = 0.999

6908 measured reflections

6542 independent reflections

2954 observed reflections

[*I* > 3.00σ(*I*)]

*R<sub>int</sub>* = 0.021

θ<sub>max</sub> = 77.88°

*h* = 0 → 12

*k* = 0 → 19

*l* = –24 → 24

3 standard reflections

monitored every 200

reflections

intensity decay: 0.7%

### Refinement

Refinement on *F*

*R* = 0.039

*wR* = 0.035

*S* = 1.85

2954 reflections

389 parameters

H-atom parameters not refined

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*)]

(Δ/σ)<sub>max</sub> = 0.002

Δρ<sub>max</sub> = 0.11 e Å<sup>-3</sup>

Δρ<sub>min</sub> = –0.15 e Å<sup>-3</sup>

Extinction correction:

Zachariasen (1968)

Extinction coefficient:

0.00000157

Atomic scattering factors

from International Tables for Crystallography (1992, Vol. C), Tables 4.2.6.8

and 6.1.1.1 (C, O), Table 6.1.1.2 (H)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
O(1)	0.9551 (1)	0.06157 (9)	0.77240 (8)	0.0485 (5)
C(1)	0.5313 (3)	0.2606 (2)	0.8100 (2)	0.0697 (10)
C(2)	0.5288 (3)	0.3080 (2)	0.8704 (2)	0.086 (1)
C(3)	0.6392 (4)	0.3536 (2)	0.8963 (2)	0.087 (1)
C(4)	0.7558 (3)	0.3541 (2)	0.8619 (2)	0.071 (1)
C(4a)	0.7586 (3)	0.3075 (2)	0.8021 (1)	0.0564 (9)
C(5)	0.8821 (3)	0.3838 (2)	0.6461 (2)	0.0659 (10)
C(6)	0.8277 (3)	0.3954 (2)	0.5786 (2)	0.079 (1)
C(7)	0.7221 (3)	0.3464 (2)	0.5505 (2)	0.076 (1)
C(8)	0.6659 (3)	0.2860 (2)	0.5911 (2)	0.0663 (10)
C(8a)	0.7185 (2)	0.2747 (2)	0.6591 (1)	0.0533 (8)
C(9)	0.6714 (2)	0.2109 (1)	0.7106 (1)	0.0522 (8)
C(9a)	0.6469 (2)	0.2609 (2)	0.7758 (1)	0.0548 (8)
C(10)	0.8795 (2)	0.2974 (1)	0.7596 (1)	0.0532 (8)
C(10a)	0.8288 (2)	0.3221 (2)	0.6857 (1)	0.0534 (8)
C(11)	0.9020 (2)	0.2024 (1)	0.7572 (1)	0.0478 (7)
C(12)	1.0198 (2)	0.1445 (1)	0.7767 (1)	0.0454 (7)
C(13)	0.8183 (2)	0.0657 (1)	0.7377 (1)	0.0452 (7)
C(14)	0.7958 (2)	0.1590 (1)	0.7328 (1)	0.0466 (7)
C(15)	1.1240 (2)	0.1503 (2)	0.7239 (1)	0.0472 (7)
C(16)	1.1782 (3)	0.2278 (2)	0.7093 (1)	0.0597 (9)
C(17)	1.2738 (3)	0.2352 (2)	0.6622 (1)	0.070 (1)
C(18)	1.3151 (3)	0.1649 (2)	0.6293 (1)	0.076 (1)
C(19)	1.2641 (3)	0.0877 (2)	0.6437 (2)	0.076 (1)
C(20)	1.1683 (3)	0.0800 (2)	0.6908 (1)	0.0625 (9)
C(21)	1.0842 (2)	0.1546 (1)	0.8506 (1)	0.0495 (8)
C(22)	1.2175 (3)	0.1337 (2)	0.8676 (1)	0.0613 (9)
C(23)	1.2735 (3)	0.1417 (2)	0.9351 (2)	0.082 (1)
C(24)	1.1991 (4)	0.1711 (2)	0.9860 (2)	0.089 (1)
C(25)	1.0660 (4)	0.1916 (2)	0.9699 (2)	0.082 (1)
C(26)	1.0081 (3)	0.1830 (2)	0.9024 (2)	0.0647 (9)
C(27)	0.8199 (2)	0.0205 (2)	0.6686 (1)	0.0495 (8)
C(28)	0.8384 (3)	0.0637 (2)	0.6085 (2)	0.071 (1)
C(29)	0.8499 (3)	0.0205 (3)	0.5471 (2)	0.093 (1)
C(30)	0.8429 (3)	–0.0660 (3)	0.5458 (2)	0.092 (1)
C(31)	0.8239 (3)	–0.1094 (2)	0.6050 (2)	0.080 (1)
C(32)	0.8120 (3)	–0.0661 (2)	0.6660 (1)	0.0625 (9)
C(33)	0.7197 (2)	0.0278 (1)	0.7847 (1)	0.0494 (8)
C(34)	0.7522 (3)	0.0203 (2)	0.8552 (2)	0.0649 (9)
C(35)	0.6559 (4)	–0.0067 (2)	0.8978 (2)	0.085 (1)
C(36)	0.5285 (4)	–0.0265 (2)	0.8697 (2)	0.098 (1)
C(37)	0.4957 (3)	–0.0204 (2)	0.8000 (2)	0.090 (1)
C(38)	0.5907 (3)	0.0071 (2)	0.7573 (1)	0.0675 (10)

Table 2. Selected geometric parameters (Å, °)

O(1)—C(12)	1.465 (2)	C(11)—C(14)	1.314 (3)
O(1)—C(13)	1.466 (2)	C(13)—C(14)	1.499 (3)
C(11)—C(12)	1.514 (3)		
C(12)—O(1)—C(13)	111.8 (2)	C(1)—C(9a)—C(9)	126.6 (3)
C(4)—C(4a)—C(10)	126.0 (3)	C(5)—C(10a)—C(10)	126.3 (3)
C(8)—C(8a)—C(9)	126.6 (2)		

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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## C<sub>20</sub>H<sub>10</sub>F<sub>8</sub>, a Divinyl Acetylene

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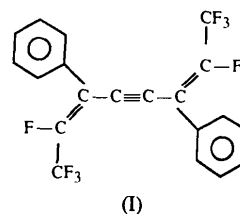
### Abstract

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 CF<sub>3</sub> 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.

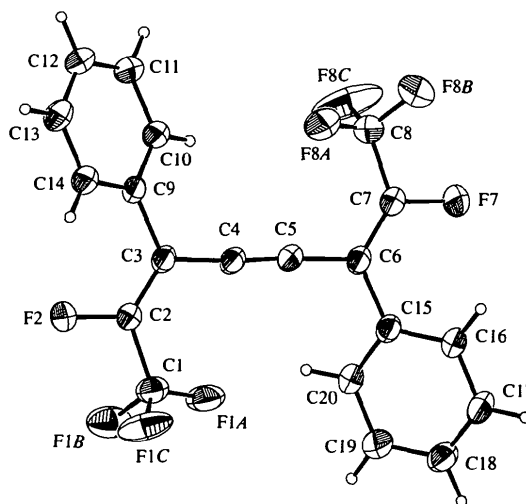


Fig. 1. View and numbering scheme of the title compound. Ellipsoids are drawn at the 35% level. Only one orientation of each disordered trifluoromethyl group is shown for clarity. H atoms are given an arbitrarily small radius.

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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