$C_{18}H_{14}ClN_3O$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.080$

$S = 1.037$

3676 reflections

210 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0390P)^2 + 0.1372P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.147$ e Å^{-3}

$\Delta\rho_{min} = -0.160$ e Å^{-3}

Extinction correction: SHELXL97

Extinction coefficient: 0.018 (1)

Scattering factors from International Tables for Crystallography (Vol. C)

Absolute structure: Flack (1983)

Flack parameter = 0.03 (5)

Table 1. *Selected geometric parameters (Å, °)*

C11—C4''	1.7423 (15)	N1—C1''	1.4311 (18)
O1—C4'	1.3761 (18)	N2—C3	1.3377 (18)
O1—C7'	1.423 (2)	N3—C7	1.132 (3)
N1—C5	1.363 (2)	C3—C4	1.407 (2)
N1—N2	1.3650 (17)	C4—C5	1.366 (2)
C5—N1—N2	111.97 (12)	N1—C5—C4	106.50 (14)
C3—N2—N1	104.66 (12)	N1—C5—C6	121.71 (13)
N2—C3—C4	110.97 (13)	N3—C7—C6	179.3 (2)
C5—C4—C3	105.90 (14)		
N1—C5—C6—C7	179.27 (16)	C7'—O1—C4'—C3'	170.19 (17)
N2—C3—C1'—C6'	−6.6 (2)	N2—N1—C1''—C2''	57.7 (2)

The temperature of the crystal was controlled using an Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). Data were collected over a hemisphere of reciprocal space, by a combination of three sets of exposures. Each set had a different φ angle for the crystal and each exposure of 10 s covered $0.3°$ in ω. The crystal-to-detector distance was 5.01 cm. Coverage of the unique set was over 93% complete to at least $28°$ in θ. Crystal decay was monitored by repeating the initial frames at the end of the data collection and analysing the duplicate reflections; it was found to be negligible. H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of their parent atoms.

Data collection: *SMART* (Siemens, 1994a). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL/PC* (Siemens, 1994b). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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3,4,5,6-Tetrafluoro-1,2,7,8-tetrakis(trifluoromethyl)phenanthrene and 3,4,7,8-Tetrafluoro-1,2,5,6-tetrakis(trifluoromethyl)anthracene

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Abstract

A photocyclization reaction produces two isomers of $C_{18}H_2F_{16}$. Crystal structures show the major product to be 3,4,5,6-tetrafluoro-1,2,7,8-tetrakis(trifluoromethyl)phenanthrene, (1), and the minor product to be 3,4,7,8-tetrafluoro-1,2,5,6-tetrakis(trifluoromethyl)anthracene, (2). The phenanthrene skeleton in (1) is distorted from planarity by steric interaction between the 4- and 7-F atoms. Compound (2) is centered on an inversion center; the anthracene skeleton is nearly planar,

with the trifluoromethyl atoms displaced on either side of the molecular plane.

Comment

Photocyclization of (Z)-1,1,2,5,5,5-hexafluoro-4-{p-[(Z)-1,2-bis(trifluoromethyl)-3,4,4-trifluoro-1,3-butadiene]-phenyl}-3-trifluoromethyl-1,3-pentadiene at 254 nm gives a mixture of isomers, (1) and (2) (see *Experimental*), which were separated *via* elution over silica gel with hexane. The predominant isomer is (1). X-ray diffraction analyses establish the structures of both isomers.

(1)   (2)
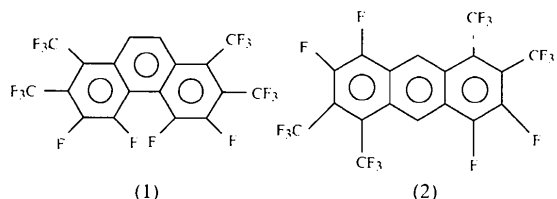
The central ring of (1) (C5, C6, C11–C14) deviates significantly from planarity, presumably due to the F4···F7 close contact (Fig. 1*a*). The largest deviation from planarity for the C1–C5, C14 ring (plane 1) is 0.058 (2) Å for C14. The largest deviation from planarity for the C6–C11 ring (plane 2) is 0.064 (2) Å for C11. The dihedral angle between planes 1 and 2 is 27.3 (1)°.
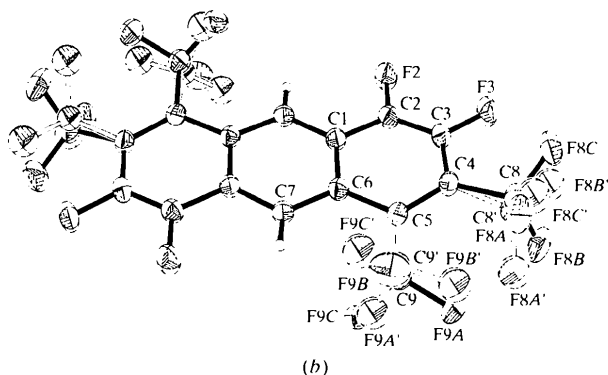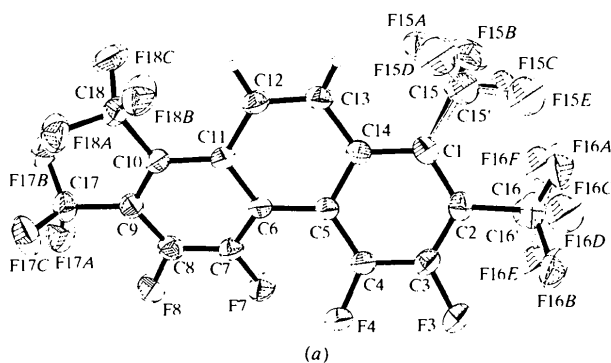

(a)

(b)

Fig. 1. Views of (*a*) molecule (1) and (*b*) molecule (2) showing displacement ellipsoids at the 35% probability level. Open bonds represent the minor component of the disordered trifluoromethyl groups.

The central ring has a slight twist conformation. The trifluoromethyl groups at C1 and C2, and also at C9 and C10 are situated slightly above and below planes 1 and 2, respectively (torsion-angle data and selected non-bonding F···F distances are in Table 2).

There is a minor disorder in the positioning of the C15 and C16 trifluoromethyl groups. The minor conformation [11.3 (3)%] has the trifluoromethyl groups flipped to the other side of plane 1. The molecules of (1) stack in a tilted manner along the unit-cell **c** direction. The stacks pack with a herring-bone motif.

Molecules of (2) (Fig. 1*b*) are centered on crystallographic inversion centers and stack in a tilted manner along the unit-cell **a** direction. A minor disorder [5.4 (2)%] in the trifluoromethyl conformation similar to that found in (1) also exists in (2).

Atoms C4 and C5 in (2) are the sites of the trifluoromethyl substitution and have the largest deviation [0.046 (1) and −0.063 (1) Å, respectively] from the molecular least-squares plane. The C8 and C9 methyl C atoms are 0.274 (3) and −0.277 (2) Å above and below the molecular plane. Presumably, these distortions from planarity are induced by steric interaction between the adjacent trifluoromethyl groups (torsion-angle data and selected non-bonding F···F contacts are in Table 4).

Experimental

(Z)-1,1,2,5,5,5-Hexafluoro-4-{p-[(Z)-1,2-bis(trifluoromethyl)-3,4,4-trifluoro-1,3-butadiene]phenyl}-3-trifluoromethyl-1,3-pentadiene (1 g) was dissolved in hexane (10 ml) in a Rotaflo quartz tube. After freezing this solution with liquid nitrogen, the tube was degassed with a vacuum pump. The solution was thawed and placed in the photoreactor (254 nm) at room temperature overnight. After reaction, the solvent was evaporated and the residue obtained was purified *via* silica-gel (70–230 mesh) chromatography (hexane) to give (1) and (2) with 28 and 7% yields, respectively. Crystals were grown from hexane–benzene by slow evaporation.

Compound (1)

Crystal data

$C_{18}H_2F_{16}$	Mo $K\alpha$ radiation
$M_r = 522.20$	$\lambda = 0.7107$ Å
Monoclinic	Cell parameters from 22
$P2_1$	reflections
$a = 8.291\,(1)$ Å	$\theta = 10.5–15.1°$
$b = 26.735\,(5)$ Å	$\mu = 0.239$ mm^{-1}
$c = 8.567\,(1)$ Å	$T = 293\,(2)$ K
$\beta = 114.49\,(2)°$	Prism
$V = 1728.1\,(4)$ Å^3	$0.55 \times 0.36 \times 0.26$ mm
$Z = 4$	Colorless
$D_x = 2.007$ Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.022$
diffractometer	$\theta_{max} = 27.5°$

$\theta/2\theta$ scans
Absorption correction: none
8254 measured reflections
3816 independent reflections
2639 reflections with
 $I > 2\sigma(I)$

$h = -10 \rightarrow 7$
$k = -24 \rightarrow 34$
$l = -11 \rightarrow 10$
4 standard reflections
 frequency: 240 min
 intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.140$
$S = 1.100$
3815 reflections
329 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2$
 $+ 1.091P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.025$
$\Delta\rho_{max} = 0.319$ e Å^{-3}
$\Delta\rho_{min} = -0.242$ e Å^{-3}
Extinction correction:
 SHELXTL
Extinction coefficient:
 0.0186 (17)
Scattering factors from
 *International Tables for
 Crystallography* (Vol. C)

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

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

	x	y	z	U_{eq}
C1	−0.2391 (3)	0.18451 (9)	−0.4915 (3)	0.0458 (6)
C2	−0.4237 (3)	0.18513 (10)	−0.5583 (3)	0.0485 (6)
C3	−0.5039 (3)	0.15946 (10)	−0.4687 (3)	0.0488 (6)
F3	−0.6812 (2)	0.15738 (7)	−0.5254 (2)	0.0699 (5)
C4	−0.4084 (3)	0.13521 (9)	−0.3177 (3)	0.0427 (5)
F4	−0.5021 (2)	0.11007 (6)	−0.2491 (2)	0.0587 (4)
C5	−0.2234 (3)	0.13368 (8)	−0.2457 (3)	0.0372 (5)
C6	−0.1159 (3)	0.10924 (8)	−0.0834 (3)	0.0371 (5)
C7	−0.1683 (3)	0.10299 (10)	0.0502 (3)	0.0443 (5)
F7	−0.3150 (2)	0.12471 (7)	0.0463 (2)	0.0630 (5)
C8	−0.0663 (3)	0.07773 (11)	0.1977 (3)	0.0487 (6)
F8	−0.1322 (2)	0.07473 (8)	0.3163 (2)	0.0728 (5)
C9	0.0955 (3)	0.05640 (9)	0.2230 (3)	0.0447 (5)
C10	0.1552 (3)	0.06284 (9)	0.0934 (3)	0.0427 (5)
C11	0.0579 (3)	0.09267 (9)	−0.0522 (3)	0.0393 (5)
C12	0.1325 (3)	0.11157 (11)	−0.1652 (3)	0.0496 (6)
C13	0.0454 (3)	0.14379 (11)	−0.2917 (3)	0.0510 (6)
C14	−0.1392 (3)	0.15557 (9)	−0.3434 (3)	0.0399 (5)
C15†	−0.1353 (5)	0.21765 (14)	−0.5614 (5)	0.0636 (9)
F15A†	0.0135 (3)	0.23526 (9)	−0.4359 (3)	0.0851 (8)
F15B†	−0.0840 (4)	0.19457 (12)	−0.6699 (3)	0.0928 (9)
F15C†	−0.2243 (4)	0.25845 (10)	−0.6343 (4)	0.1076 (11)
C15′‡	−0.155 (2)	0.2092 (6)	−0.6017 (2)	0.064
F15D‡	−0.043 (3)	0.1767 (7)	−0.617 (3)	0.095
F15E‡	−0.267 (2)	0.2245 (8)	−0.754 (2)	0.095
F15F‡	−0.067 (3)	0.2478 (8)	−0.510 (2)	0.095
C16†	−0.5424 (5)	0.20824 (15)	−0.7309 (4)	0.0644 (9)
F16A†	−0.4712 (4)	0.20652 (11)	−0.8437 (3)	0.1005 (9)
F16B†	−0.6956 (3)	0.18380 (11)	−0.8049 (3)	0.0984 (9)
F16C†	−0.5798 (4)	0.25518 (9)	−0.7164 (3)	0.0894 (8)
C16′‡	−0.549 (2)	0.2237 (4)	−0.684 (2)	0.064
F16D‡	−0.587 (3)	0.2032 (7)	−0.835 (3)	0.097
F16E‡	−0.697 (2)	0.2325 (7)	−0.666 (2)	0.097
F16F‡	−0.464 (2)	0.2663 (6)	−0.672 (3)	0.097
C17	0.2045 (4)	0.03071 (11)	0.3948 (3)	0.0573 (7)
F17A	0.1593 (3)	0.04611 (9)	0.5176 (2)	0.0839 (6)
F17B	0.3762 (2)	0.04166 (7)	0.4493 (2)	0.0728 (5)
F17C	0.1874 (3)	−0.01866 (7)	0.3838 (3)	0.0847 (6)
C18	0.3220 (4)	0.03747 (12)	0.0999 (4)	0.0569 (7)
F18A	0.3590 (3)	−0.00490 (7)	0.1890 (3)	0.0769 (6)
F18B	0.3038 (3)	0.02343 (8)	−0.0568 (3)	0.0822 (6)
F18C	0.4630 (2)	0.06655 (8)	0.1645 (3)	0.0860 (6)

† Site occupancy = 0.887 (3).   ‡ Site occupancy = 0.113 (3).

Table 2. *Selected geometric parameters (Å, °) for (1)*

C3—F3	1.344 (3)	C12—C13	1.337 (4)
C4—F4	1.334 (3)	F3···F4	2.548 (2)
C7—F7	1.336 (3)	F4···F7	2.390 (2)
C8—F8	1.341 (3)	F7···F8	2.554 (2)
C5—C4—F4	121.7 (2)	C5—C6—C7	124.2 (2)
C4—C5—C6	124.0 (2)	C6—C7—F7	121.2 (2)
C14—C1—C2—C3	4.3 (4)	C8—C9—C10—C11	3.8 (4)
C15—C1—C2—C16	15.5 (4)	C17—C9—C10—C18	10.1 (4)
C4—C5—C6—C7	−27.6 (4)	C11—C12—C13—C14	−10.7 (4)

Compound (2)

Crystal data

$C_{18}H_2F_{16}$
$M_r = 522.20$
Triclinic
$P\bar{1}$
$a = 8.268$ (1) Å
$b = 8.469$ (2) Å
$c = 8.274$ (2) Å
$\alpha = 126.06$ (2)°
$\beta = 110.62$ (2)°
$\gamma = 90.19$ (2)°
$V = 421.0$ (2) Å^3
$Z = 1$
$D_x = 2.060$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25
 reflections
$\theta = 14.0$–22.0°
$\mu = 0.245$ mm^{-1}
$T = 291$ (2) K
Thick plate
0.44 × 0.37 × 0.22 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4
 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3758 measured reflections
1915 independent reflections
1603 reflections with
 $I > 2\sigma(I)$

$R_{int} = 0.019$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -10 \rightarrow 9$
4 standard reflections
 frequency: 240 min
 intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.117$
$S = 1.110$
1914 reflections
172 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2$
 $+ 0.1566P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.100$
$\Delta\rho_{max} = 0.267$ e Å^{-3}
$\Delta\rho_{min} = -0.206$ e Å^{-3}
Extinction correction:
 SHELXTL
Extinction coefficient:
 0.042 (8)
Scattering factors from
 *International Tables for
 Crystallography* (Vol. C)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (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}
C1	0.9369 (2)	−0.0771 (2)	0.0814 (3)	0.0353 (3)
C2	0.8727 (2)	−0.1519 (3)	0.1681 (3)	0.0422 (4)
F2	0.9110 (2)	−0.3185 (2)	0.1372 (2)	0.0609 (4)
C3	0.7775 (2)	−0.0576 (3)	0.2855 (3)	0.0440 (4)
F3	0.7236 (2)	−0.1384 (2)	0.3632 (2)	0.0641 (4)
C4	0.7304 (2)	0.1186 (3)	0.3214 (3)	0.0397 (4)
C5	0.7740 (2)	0.1853 (2)	0.2209 (3)	0.0370 (4)
C6	0.8864 (2)	0.0954 (2)	0.1075 (3)	0.0347 (3)
C7	0.9542 (2)	0.1697 (2)	0.0248 (3)	0.0374 (4)

C8†	0.6434 (3)	0.2291 (3)	0.4812 (3)	0.0487 (5)
F8A†	0.7035 (2)	0.4299 (2)	0.6055 (2)	0.0646 (4)
F8B†	0.4656 (2)	0.1764 (2)	0.3769 (3)	0.0704 (5)
F8C†	0.6841 (2)	0.1906 (3)	0.6228 (3)	0.0761 (5)
C8'‡	0.609 (2)	0.212 (3)	0.440 (3)	0.049
F8A'‡	0.473 (3)	0.236 (4)	0.320 (4)	0.073
F8B'‡	0.548 (4)	0.087 (3)	0.463 (4)	0.073
F8C'‡	0.703 (3)	0.390 (4)	0.637 (4)	0.073
C9†	0.7027 (3)	0.3492 (3)	0.2174 (3)	0.0463 (5)
F9A†	0.5545 (2)	0.3710 (3)	0.2485 (3)	0.0730 (5)
F9B†	0.8258 (3)	0.5289 (2)	0.3679 (3)	0.0760 (5)
F9C†	0.6542 (2)	0.3049 (3)	0.0198 (2)	0.0607 (4)
C9'‡	0.752 (3)	0.388 (2)	0.270 (4)	0.046
F9A'‡	0.646 (4)	0.319 (5)	0.066 (3)	0.070
F9B'‡	0.672 (3)	0.487 (4)	0.400 (4)	0.070
F9C'‡	0.904 (3)	0.514 (4)	0.347 (5)	0.070

† Site occupancy = 0.946 (2).   ‡ Site occupancy = 0.054 (2).

Table 4. *Selected geometric parameters* ($\mathring{A}$, °) *for* (2)

C2—F2	1.339 (2)	F2···F3	2.631 (2)
C3—F3	1.341 (2)		
C3—C4—C5—C6	−8.4 (2)	C8—C4—C5—C9	−14.0 (3)

The H atoms in each structure were refined with independent isotropic displacement parameters. The C15 and C16 CF$_3$ groups in (1), and the C8 and C9 CF$_3$ groups in (2) were slightly disordered. In each case, the atoms of the major component of the disorder were refined with anisotropic displacement parameters. The occupancy for the major component in (1) and (2) refined to 0.887 (3) and 0.946 (2), respectively. In each case, the sum of the occupancy of the major plus minor component was constrained to be 1.0. In both structures, the minor disorder component CF$_3$ groups were treated as rigid groups; C—F 1.32 $\mathring{A}$, tetrahedral angles, $U_{iso}(C') = U_{iso}(C)$ and $U_{iso}(F') = 1.5U_{iso}(C')$. The ring–carbon distances were restrained to be the same (*i.e.* C1—C15 = C1—C15', *etc.*; s.u. = 0.01 $\mathring{A}$)

For both compounds, data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977); cell refinement: *CAD-4 Operations Manual*; data reduction: *PROCESS* in *MolEN* (Fair, 1990); program(s) used to solve structures: *MULTAN*80 (Main *et al.*, 1980); program(s) used to refine structures: *SHELXTL* (Sheldrick, 1995); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Ethyl 3,3-Dimethyl-2-(1-oxo-3-phenylinden-2-yl)acrylate

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Abstract

The crystal structure of the title compound, C$_{22}$H$_{20}$O$_3$, was determined from intensities collected at 120 K. The dihedral angle between the phenyl ring and the indenone moiety is 124.6 (1)°. The non-bonded distance between the potentially reacting centers is 3.697 (5) $\mathring{A}$.

Comment

The title compound, (I), was synthesized (Heller, 1985) as a representative of a new potentially photochromic system that is similar to the fulgides (Stobbe & Eckert, 1905; Stobbe, 1911; Heller & Langan, 1981; Heller & Oliver, 1981). It was hoped that the photochemical ring closure from (I) to (II) would be possible.
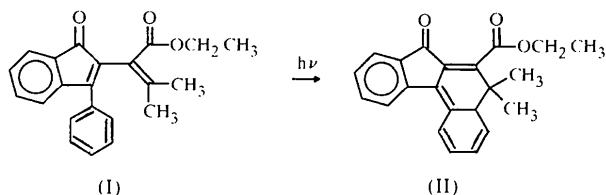
The crystal structure of (I) was determined at low temperature (120 K); the conformation of the butadiene fragment is compared with that of some related fulgides (Kaftory *et al.*, 1998).

Some of the fulgides (Kaftory *et al.*, 1998) are very rigid and because of the bulky substituents at the double bonds, the diene system in these compounds is severely skewed. In contrast, the diene system in (I) is not rigid and free rotation about the C1—C2 bond determines the conformation of the molecule. The torsion angle of the butadienyl fragment, C2—C1—C16—C17, is −63.1 (5)° in (I), while it is 55 (1)° in 2-fluorenylidene-3-adamantylidenesuccinic anhydride and 39 (1)° in 2-fluorenylidene-3-(3,4,5-trimethoxybenzylidene)succinic anhydride, as found by Kaftory *et al.* (1998).

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