

of the unit cell, in contrast with its distribution on both sides of the origin in $(TMTTF)_2BF_4$ and $(TMTSF)_2BF_4$.

References

- AUBAN, P., JÉRÔME, D., LERSTRUP, K., JOHANNSEN, I., JORGENSEN, M. & BECHGAARD, K. (1989). *J. Phys. (Paris)*, **50**, 2727–2739.
 B. A. FRENZ & ASSOCIATES, INC. (1985). *SDP Structure Determination Package*. College Station, Texas, USA.
 BECHGAARD, K., JACOBSEN, J., MORTENSEN, K., PEDERSEN, H. J. & THORUP, N. (1980). *Solid State Commun.* **33**, 1119–1125.

- BRUN, G., LIAUTARD, B., PEYTAVIN, S., MAURIN, M., TOREILLES, E., FABRE, J. M., GIRAL, L. & GALIGNÉ, J. L. (1977). *J. Phys. (Paris) Colloq.* **38**, 266–269.
 GALIGNÉ, J. L., LIAUTARD, B., PEYTAVIN, S., BRUN, G., MAURIN, M., FABRE, M., TORREILLES, E. & GIRAL, L. (1979). *Acta Cryst.* **B35**, 1129–1135.
 KOBAYASHI, H., KOBAYASHI, A., SAITO, G. & INOKUCHI, H. (1982). *Chem. Soc. Jpn, Chem. Lett.* pp. 245–248.
 LERSTRUP, K., JOHANNSEN, I. & JORGENSEN, M. (1988). *Synth. Met.* **27**, B9–B13.
 TRIKI, S., OUAHAB, L., GRANDJEAN, D., AMOUROUX, J. & FABRE, J. M. (1991). *Acta Cryst.* **C47**, 1941–1943.
 WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1991). **C47**, 1982–1983

3',3',4',4'-Tetrafluoro-4-iodo-1-phenyl-2,3-cyclobutenonaphthalene

BY M. G. BARLOW, R. G. PRITCHARD, S. TAJAMMAL AND A. E. TIPPING

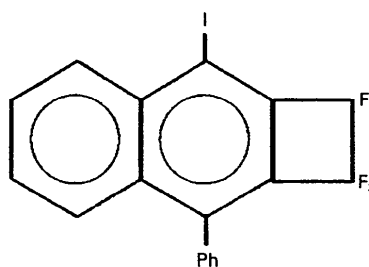
Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

(Received 6 December 1990; accepted 13 February 1991)

Abstract. $C_{18}H_9F_4I$, $M_r = 428.2$, orthorhombic, *Pbca*, $a = 20.039$ (7), $b = 18.800$ (9), $c = 8.167$ (3) Å, $V = 3077$ Å³, $Z = 8$, $D_x = 1.848$ Mg m⁻³, $F(000) = 1648$, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 2.09$ mm⁻¹, $T = 293$ K, $R = 0.034$ for 1555 unique reflexions [$F \geq 3\sigma(F)$]. The title compound is a by-product (up to 11.5%) in the synthesis of 3,3-difluoro-3-iodo-1-phenylpropyne, $Ph-C\equiv C-CF_2I$. It would have been formed by dimerization of the latter *via* elimination of iodine to give 1,6-diphenyltetrafluoro-hexa-1,5-diyne, $Ph-C\equiv C-CF_2-CF_2-C\equiv C-Ph$, followed by a double cyclization, induced by electrophilic attack by iodine on a carbon adjacent to a phenyl ring. The planar naphthalene moiety retains a bond shortening pattern typical of its class and is inclined at 54 (1)° to the phenyl ring. This ring is directed away from a C—I bond, which at 2.094 (6) Å falls within the expected range.

Experimental. Sample preparation involved treating a solution of 3-bromo-3,3-difluoro-1-phenylpropyne (0.088 mol in 100 ml acetone) with an excess of sodium iodide (0.367 mol in 200 ml acetone) followed by refluxing in a nitrogen atmosphere for 35 h. The resulting solution was partitioned between *n*-hexane and water prior to drying the organic layer over $MgSO_4$. The solvent was then removed using a rotary evaporator and the crude product separated into its three components using dry-column flash chromatography (70/55 mm sinter packed with Kieselgel silica eluted with petroleum ether, b.p.

313–333 K). The products in order of elution were (i) 3,3-difluoro-3-iodo-1-phenylpropyne (R_F 0.57; 60%), identified by spectral (¹H, ¹³C and ¹⁹F NMR and mass) data and elemental analysis, (ii) (*E*)-1-bromo-1,2-diiido-2-phenylethene (R_F 0.42; ca 1%), formed by addition of iodine across the triple bond of 1-bromo-2-phenylethyne impurity in the reactant and identified by single-crystal X-ray crystallography (Barlow, Tajammal & Tipping, 1991), and (iii) the title compound (R_F 0.33; 11.5%).



Crystal dimensions 0.10 × 0.15 × 0.25 mm, Rigaku AFC6S diffractometer, graphite-monochromated $Mo K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centered reflexions ($12.2 \leq 2\theta \leq 18.7^\circ$), ω - 2θ scan mode, ω -scan width ($1.15 + 0.30 \tan \theta$)° and scan speed of 8° min⁻¹ with up to two additional scans of weak reflexions [$I < 10\sigma(I)$], $0 \leq h \leq 23$, $0 \leq k \leq 22$, $-9 \leq l \leq 0$, $0 \leq \theta \leq 25^\circ$, 3108 unique reflexions measured, 1555 observed [$I \geq$

Table 1. *Final coordinates and B_{eq} (\AA^2) for non-H atoms*

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

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
I(1)	0.54419 (3)	0.68924 (2)	0.63657 (7)	4.86 (3)
F(11A)	0.2867 (2)	0.5797 (3)	0.8806 (7)	7.6 (3)
F(11B)	0.2775 (2)	0.5647 (2)	0.6226 (7)	7.2 (3)
F(12A)	0.3641 (3)	0.6951 (2)	0.8339 (7)	7.4 (3)
F(12B)	0.3512 (2)	0.6831 (2)	0.5727 (7)	7.5 (3)
C(1)	0.4116 (3)	0.4717 (3)	0.7640 (8)	3.0 (3)
C(2)	0.3871 (3)	0.5383 (3)	0.7385 (9)	3.3 (3)
C(3)	0.4242 (4)	0.5985 (3)	0.7085 (9)	3.6 (3)
C(4)	0.4915 (4)	0.5962 (3)	0.6925 (8)	3.2 (3)
C(5)	0.5930 (3)	0.5203 (4)	0.7072 (10)	3.9 (4)
C(6)	0.6232 (4)	0.4568 (4)	0.7324 (12)	5.1 (4)
C(7)	0.5842 (4)	0.3968 (4)	0.7631 (12)	5.1 (5)
C(8)	0.5167 (4)	0.4022 (4)	0.7717 (10)	3.8 (4)
C(9)	0.4828 (3)	0.4677 (3)	0.7510 (8)	2.7 (3)
C(10)	0.5233 (3)	0.5296 (3)	0.7178 (8)	3.1 (3)
C(11)	0.3220 (4)	0.5795 (4)	0.7414 (11)	4.8 (4)
C(12)	0.3652 (4)	0.6468 (4)	0.7110 (11)	5.0 (4)
C(13)	0.3675 (3)	0.4104 (3)	0.7968 (8)	3.0 (3)
C(14)	0.3163 (4)	0.3943 (4)	0.6924 (9)	3.7 (3)
C(15)	0.2745 (4)	0.3374 (4)	0.7267 (11)	4.5 (4)
C(16)	0.2824 (4)	0.2970 (4)	0.8626 (12)	4.5 (4)
C(17)	0.3321 (4)	0.3130 (4)	0.9671 (10)	4.3 (4)
C(18)	0.3755 (3)	0.3683 (4)	0.9368 (9)	3.5 (3)

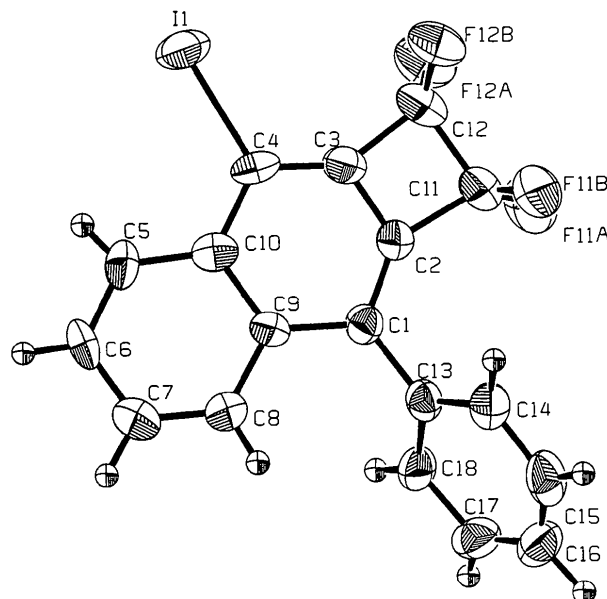
$3\sigma(I)$; intensity standards ($4\bar{1}2$; $\bar{1}13$; $\bar{1}6\bar{1}$) measured after every 150 reflexions showed no systematic drift. L_p absorption (max., min. transmission 1.0, 0.78) and secondary-extinction corrections applied (extinction coefficient 0.98491×10^{-7}). *MITHRIL* (Gilmore, 1984) used to solve the phase problem, all non-H atoms found in Fourier map, H atoms from ΔF synthesis. Full-matrix least squares based on F using the *TEXSAN* crystallographic software (Molecular Structure Corporation, 1985). Final $R = 0.034$, $wR = 0.040$, $w = 1/[\sigma^2(F_o) + (0.03F_o)^2]$ for 245 variables, anisotropic thermal parameters for heavier atoms, isotropic for H atoms. Maximum fluctuation in final ΔF map in range -0.56 to 0.42 e \AA^{-3} , max. Δ/σ 0.004. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2A); computation carried out on a Digital VAX station 3520. Literature survey performed via the Cambridge Structural Database using the Crystal Structure Search and Retrieval interactive system (CSSR, 1984). Fractional atomic coordinates and vibrational parameters for non-H atoms are presented in Table 1* and selected bond lengths and angles in Table 2. The title molecule, including atomic labelling, is displayed in Fig. 1.

Related literature. The molecular dimensions reported here show good agreement with those in

* Lists of structure factors, H-atom coordinates and anisotropic vibrational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53996 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected bond lengths (\AA) and angles ($^\circ$)*

I(1)—C(4)	2.094 (6)	C(5)—C(6)	1.35 (1)
F(11A)—C(11)	1.339 (9)	C(5)—C(10)	1.41 (1)
F(11B)—C(11)	1.348 (9)	C(6)—C(7)	1.39 (1)
F(12A)—C(12)	1.354 (8)	C(7)—C(8)	1.36 (1)
F(12B)—C(12)	1.350 (9)	C(8)—C(9)	1.416 (8)
C(1)—C(2)	1.360 (8)	C(9)—C(10)	1.446 (7)
C(1)—C(9)	1.433 (8)	C(11)—C(12)	1.55 (1)
C(1)—C(13)	1.477 (8)	C(13)—C(14)	1.368 (9)
C(2)—C(3)	1.376 (9)	C(13)—C(18)	1.399 (9)
C(2)—C(11)	1.518 (9)	C(14)—C(15)	1.38 (1)
C(3)—C(4)	1.355 (9)	C(15)—C(16)	1.35 (1)
C(3)—C(12)	1.490 (9)	C(16)—C(17)	1.35 (1)
C(4)—C(10)	1.420 (9)	C(17)—C(18)	1.377 (9)
C(2)—C(1)—C(9)	113.3 (6)	C(2)—C(11)—C(12)	86.2 (5)
C(1)—C(2)—C(3)	126.2 (7)	C(4)—C(3)—C(12)	144.1 (7)
C(1)—C(2)—C(11)	141.0 (6)	C(3)—C(4)—C(10)	117.5 (6)
C(3)—C(2)—C(11)	92.7 (5)		

Fig. 1. The title molecule drawn using *ORTEPII* (Johnson, 1976).

unsubstituted naphtho[*b*]cyclobutene (Crawford & Marsh, 1973).

We thank The Committee of Vice Chancellors and Principals for an ORS Award (to ST).

References

- BARLOW, M. G., TAJAMMAL, S. & TIPPING, A. E. (1991). *J. Fluorine Chem.* In preparation.
- CRAWFORD, J. L. & MARSH, R. E. (1973). *Acta Cryst.* B29, 1238–1246.
- CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual*. SERC Daresbury Laboratory, Warrington, England.
- GILMORE, C. J. (1984). *J. Appl. Cryst.* 17, 42–46.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.
- Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX77381, USA.