Table 3. *Bond angles* (°) *with estimated standard deviations in parentheses*

O(1)S(1)–S(1)–O(2)S(1)	118·1 (5)	C(3)–C(2)–C(7)	124·6 (4)	C(5)–C(6)–H(2)C(6)	114 (1)
O(1)S(1)–S(1)–C(2)	107·0 (4)	S(4)–C(3)–C(2)	124·5 (4)	H(1)C(6)–C(6)–H(2)C(6)	109 (1)
O(2)S(1)–S(1)–C(2)	107·1 (4)	S(4)–C(3)–C(8)	111·7 (4)	C(2)–C(7)–H(1)C(7)	116 (2)
O(1)S(1)–S(1)–C(6)	109·2 (5)	C(2)–C(3)–C(8)	123·8 (4)	C(2)–C(7)–H(2)C(7)	113 (2)
O(2)S(1)–S(1)–C(6)	108·5 (4)	S(4)–C(5)–C(6)	110·1 (4)	C(2)–C(7)–H(3)C(7)	110 (2)
C(2)–S(1)–C(6)	106·3 (4)	S(4)–C(5)–H(1)C(5)	102 (1)	H(1)C(7)–C(7)–H(2)C(7)	110 (2)
O(1)S(4)–S(4)–O(2)S(4)	117·7 (3)	S(4)–C(5)–H(2)C(5)	105 (1)	H(1)C(7)–C(7)–H(3)C(7)	92 (2)
O(1)S(4)–S(4)–C(3)	108·2 (3)	C(6)–C(5)–H(1)C(5)	116 (1)	H(2)C(7)–C(7)–H(3)C(7)	114 (2)
O(2)S(4)–S(4)–C(3)	107·7 (3)	C(6)–C(5)–H(2)C(5)	114 (1)	C(3)–C(8)–H(1)C(8)	109 (2)
O(1)S(4)–S(4)–C(5)	109·3 (3)	H(1)C(5)–C(5)–H(2)C(5)	109 (1)	C(3)–C(8)–H(2)C(8)	106 (2)
O(2)S(4)–S(4)–C(5)	108·6 (3)	S(1)–C(6)–C(5)	110·3 (4)	C(3)–C(8)–H(3)C(8)	109 (2)
C(3)–S(4)–C(5)	104·7 (3)	S(1)–C(6)–H(1)C(6)	104 (1)	H(1)C(8)–C(8)–H(2)C(8)	123 (2)
S(1)–C(2)–C(3)	125·5 (4)	S(1)–C(6)–H(2)C(6)	104 (1)	H(1)C(8)–C(8)–H(3)C(8)	102 (2)
S(1)–C(2)–C(7)	109·9 (4)	C(5)–C(6)–H(1)C(6)	114 (1)	H(2)C(8)–C(8)–H(3)C(8)	107 (2)

the somewhat smaller lengthening (0·02 Å) which has been observed for vinyl–carbonyl bonds in *p*-benzoquinones when a H atom is replaced by a methyl group (Rabinovich, Schmidt & Ubell, 1967).

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5,6,7,8-Tetrafluoro-1,2,3,4-tetrahydro-9-isopropyl-1,4-methanonaphthalene

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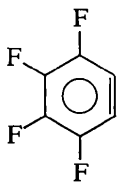
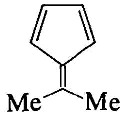
Abstract. $C_{14}H_{14}F_4$, $M_r = 258 \cdot 14$, monoclinic, $P2_1/c$ (absent reflections: $h0l$ when l odd, $0k0$ when k odd), $a = 8 \cdot 56$ (1), $b = 9 \cdot 26$ (2), $c = 15 \cdot 72$ (2) Å, $\beta = 96 \cdot 6$ (5)°, $U = 1244 \cdot 53$ Å^3, $Z = 4$, $D_x = 1 \cdot 383$ g cm^{-3}, $F(000) = 536$. The structure was solved by direct methods and refined to $R = 0 \cdot 060$ for the 806 observed reflections. The molecular configuration provides an explanation of an unexpected feature in the proton NMR spectrum.

Introduction. During a detailed study of the chemistry of arynes the reaction between tetrafluorobenzyne (I) and 6,6-dimethylfulvene (II) was studied (Hankinson, Heaney, Price & Sharma, 1973) whence the expected product 5,6,7,8-tetrafluoro-1,4-dihydro-9-isopropylidene-1,4-methanonaphthalene (III) was obtained in 48% yield. Reduction of (III) in ethanol at room temperature over palladium–carbon (5%) was terminated after the uptake of two mole equivalents of H and gave the title compound (IV) in 88% yield.



(I)



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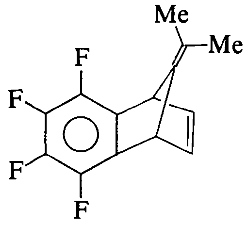

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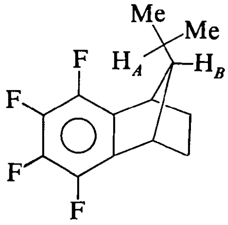

(IV)

The ^1H NMR spectrum of (IV) showed several unexpected anomalies; for example, it was expected that in a compound with the structure (IV) the isopropyl group would appear as a distinct doublet. However, at 60 MHz the methyl signal appeared to be a broadened singlet. Examination of the 220 MHz spectrum and double-irradiation experiments revealed that the methine proton H_A resonates at almost the same chemical shift as the protons in the methyl groups. The reason for this was adduced to be anisotropic shielding of H_A by the aryl residue. In order to confirm these assignments the structure of the title compound (IV) has been determined by X-ray crystallography.

Acicular crystals were grown from light petroleum. The crystal chosen for X-ray analysis was mounted in a Lindemann-glass capillary tube owing to the high volatility of the compound at ambient temperatures. Weissenberg data were collected for seven layers using Cu $K\alpha$ radiation (λ = 1·5418 Å) with the crystal rotating about the needle (a) axis. The intensities, measured by the SRC Microdensitometer Service, yielded 806 unique observed reflections. Lorentz and polarization corrections were applied but no corrections were made for absorption [μ(Cu $K\alpha$) = 11·0 cm^{-1}, crystal size 1·0 × 0·5 × 0·3 mm] or extinction.

A partial solution of the structure was achieved with $\sum_2$ relationships and a multi-symbolic phase approach using the program *PHASE* in the XRAY (1972) package. 12 atoms were located from the corresponding E map and the remaining non-hydrogen atoms were found from a subsequent Fourier map. Anisotropic refinement yielded a conventional R of 0·093. A difference map revealed the positions of all H atoms except those attached to the C(13) methyl group. Inclusion of these atoms in fixed positions, with further refinement of the original atoms, gave a final R of 0·060. In the final cycles of refinement the following weighting scheme was used: $w = 1$ for $F_o \le 20$, otherwise $w = (20/F_o)^2$. The scattering factors for C were those of Cromer & Mann (1968), those for F were taken from *International Tables for X-ray Crystallography* (1968) and for H from Stewart, Davidson &, Simpson (1965). All calculations were carried out with the XRAY (1972) system of programs as implemented at the University of Manchester Regional Computer Centre.

Table 1. *Fractional coordinates* (×10^4)

	x	y	z
C(1)	862 (9)	5645 (6)	1148 (4)
C(2)	945 (5)	4347 (12)	1591 (8)
C(3)	1722 (12)	5824 (9)	456 (6)
C(4)	1871 (5)	3266 (12)	1366 (8)
C(5)	2176 (12)	1749 (9)	1698 (7)
C(6)	2640 (6)	4724 (12)	222 (8)
C(7)	2769 (10)	3447 (9)	674 (5)
C(8)	3612 (4)	2064 (11)	575 (7)
C(9)	2362 (15)	948 (10)	231 (8)
C(10)	1365 (6)	734 (15)	987 (10)
C(11)	3892 (13)	1573 (9)	1536 (6)
C(12)	5095 (5)	2431 (13)	2109 (8)
C(13)	6732 (17)	1995 (17)	1903 (8)
C(14)	4929 (8)	2184 (23)	3069 (11)
F(1)	−27 (8)	6737 (6)	1375 (4)
F(2)	75 (4)	4226 (9)	2262 (5)
F(3)	1662 (8)	7095 (5)	47 (4)
F(4)	3452 (3)	4939 (7)	9534 (5)
H(101)	0	960	890
H(102)	1540	−430	1090
H(91)	1500	1350	−340
H(92)	2810	0	60
H(11)	4150	430	1530
H(8)	4730	2090	210
H(5)	1770	1650	2450
H(12)	5040	3480	2000
H(141)	5770	2830	3530
H(142)	5000	1000	3090
H(143)	3730	2740	3320

Table 2. *Bond lengths* (Å) *and their e.s.d.'s*

C(1)–C(2)	1·39 (1)	C(5)–C(11)	1·52 (2)
C(1)–C(3)	1·36 (1)	C(6)–C(7)	1·38 (1)
C(1)–F(1)	1·33 (1)	C(6)–F(4)	1·34 (1)
C(2)–C(4)	1·34 (1)	C(7)–C(8)	1·48 (1)
C(2)–F(2)	1·33 (1)	C(8)–C(9)	1·56 (1)
C(3)–C(6)	1·35 (1)	C(8)–C(11)	1·58 (1)
C(3)–F(3)	1·34 (1)	C(9)–C(10)	1·52 (2)
C(4)–C(5)	1·51 (1)	C(11)–C(12)	1·54 (1)
C(4)–C(7)	1·38 (1)	C(12)–C(13)	1·51 (2)
C(5)–C(10)	1·59 (2)	C(12)–C(14)	1·54 (2)

Table 3. *Bond angles* (°) *and their e.s.d.'s*

C(2)–C(1)–C(3)	119·5 (7)	C(3)–C(6)–F(4)	116·8 (10)
C(2)–C(1)–F(1)	122·3 (7)	C(7)–C(6)–F(4)	121·0 (8)
C(3)–C(1)–F(1)	118·1 (6)	C(4)–C(7)–C(6)	118·6 (8)
C(1)–C(2)–C(4)	121·7 (10)	C(4)–C(7)–C(8)	106·2 (8)
C(1)–C(2)–F(2)	117·2 (8)	C(6)–C(7)–C(8)	135·1 (9)
C(4)–C(2)–F(2)	121·1 (11)	C(7)–C(8)–C(9)	106·2 (6)
C(1)–C(3)–C(6)	118·7 (8)	C(7)–C(8)–C(11)	101·1 (8)
C(1)–C(3)–F(3)	118·9 (8)	C(9)–C(8)–C(11)	101·5 (8)
C(6)–C(3)–F(3)	122·3 (9)	C(8)–C(9)–C(10)	102·9 (9)
C(2)–C(4)–C(5)	133·9 (11)	C(5)–C(10)–C(9)	103·4 (8)
C(2)–C(4)–C(7)	119·1 (10)	C(5)–C(11)–C(8)	92·3 (7)
C(5)–C(4)–C(7)	106·9 (8)	C(5)–C(11)–C(12)	117·7 (8)
C(4)–C(5)–C(10)	104·4 (9)	C(8)–C(11)–C(12)	117·7 (8)
C(10)–C(5)–C(11)	100·9 (7)	C(11)–C(12)–C(13)	109·3 (10)
C(4)–C(5)–C(11)	101·7 (7)	C(11)–C(12)–C(14)	113·3 (9)
C(3)–C(6)–C(7)	122·2 (10)	C(13)–C(12)–C(14)	108·4 (8)

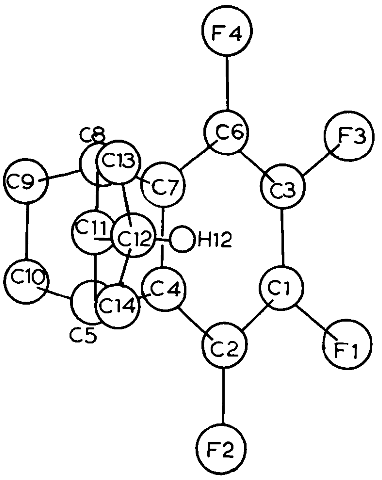

Fig. 1. Idealized molecular structure showing atom numbering.

Discussion. The final atomic coordinates are given in Table 1;* bond lengths and angles are summarized in Tables 2 and 3. The idealized molecular structure and atom numbering are shown in Fig. 1, whilst Fig. 2 shows a projection of the unit-cell contents viewed down *a*.

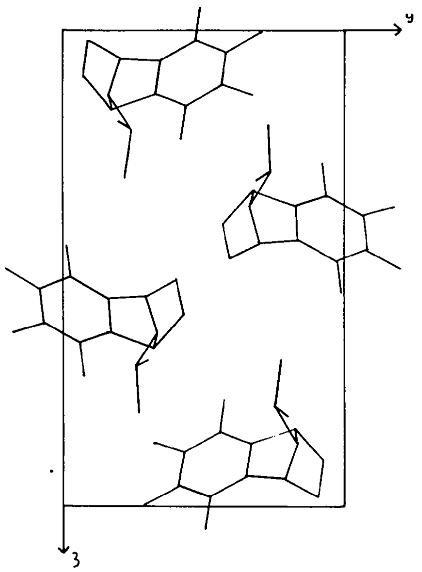

Fig. 2. Unit-cell contents projected down the *a* axis.

The structure analysis confirms that the isopropyl group attached to C(11) is pointing towards the aromatic ring, its orientation being such that the methine H atom, H(12), is directed towards the aromatic ring with the two methyl groups being oriented away. This is in accord with the interpretation of the NMR data. There are no unusual bond lengths, angles or intermolecular contacts in the structure.

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33697 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.