POLYFLUORO-COMPOUNDS BASED ON THE CYCLOHEPTANE
RING SYSTEM. PART 2. THE 1H,3H- and 1H,4H-
DODECAFLUOROCYCLOHEPTANES, DERIVED UNDECAFLUOROCYCLO-
HEPTENES AND DECAFLUOROCYCLOHEPTA-1,4- and 1,3-DIENE

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SUMMARY

    From the mixture obtained by reaction of cycloheptane with cobalt(III)
fluoride at 180-190°, the cis- and trans- 1H,3H- and 1H,4H- dodecafluoro-
cycloheptanes were isolated. Each 1H,3H- compound was dehydrofluorinated
by aqueous potash to 3H- and 4H-undecafluorocyclohept-1-ene, and thence
decafluorocyclohepta-1,4- and 1,3-diene. The 1H,4H-isomers each gave 4H-
and 5H-undecafluorocyclohept-1-ene and thence the two dienes on dehydro-
fluorination. The dichloro-adduct of the 5H-ene was reduced by lithium
aluminium hydride, and the three possible 1H,2H,5H-undecafluorocyclo-
heptanes were isolated. Likewise, the 4H-ene gave the 4 possible
1H,2H,4H-undecafluorides. All were identified by further fluorination
with cobalt(III) fluoride at 160°, to appropriate dodecafluorocycloheptanes.

RESULTS

    The mixture obtained by mild fluorination of cycloheptane by
cobalt(III) fluoride (see Part I; [1]) has been investigated further.
Previously, tridecafluorocycloheptane (T) had been isolated therefrom, and
1H/2H-(It) and 1H,2H/-dodecafluorocycloheptane(Ic) made from dodecafluoro-
cycloheptene (D), derived from compound T by dehydrofluorination (see Scheme
for structures).
    Fractional distillation of the fluorination mixture now gave the
first four fractions corresponding to those obtained before. A
significant higher-boiling fraction (5, b.r. 101.5 - 108°) was

separated by glc to give two new dodecafluorides (IIt and IIIt). The next
fraction (6, b.r. 108-114°) was difficult to process, but eventually two
further dodecafluorides (IIc and IIIc) were isolated after involved glc
separations. All four new dodecafluoro-compounds were reasonably pure,
but a few percent of impurities with CHF_2 groups, probably methylcyclo-
hexanes, were shown up by ^{19}F nmr, IIc being the least pure of them.

The dodecafluorides were subjected to dehydrofluorination studies,
paralleling earlier work on analogous polyfluorocyclo-hexanes [2] and
-pentanes [3]. Reaction with aqueous potassium hydroxide classified these
four compounds into two pairs. Both in one (IIt and IIc) gave the same
four unsaturated products (IV - VII), whilst the second pair (IIIt and
IIIc) also gave a comparable mixture of four products, three the same as
before (IV, V and VII) and one different (VIII).

Two of the dehydrofluorination products (IV and V), obtained from all
four dodecafluorides, analysed as C_7F_{10}, and showed strong absorption due
to -CF=CF- in the infrared. One, (V), had strong selective ultraviolet
absorption, attributable to conjugation; this was absent in the other (IV).
^{19}F nmr supported the allocation of structures as decafluorocyclohepta-
1,4-diene(IV), and -1,3-diene(V). Oxidation of the latter gave hexafluoro-
glutaric acid.

Each of the other three dehydrofluorination products (VI - VIII)
analysed as C_7HF_{11}, and had an appropriate strong C=C band in the infrared.
Compound VI was obtained only from IIt and IIc, and in small amount. It
was difficult to dehydrofluorinate further, but only the 1,3-diene(V) could
be detected from such reactions. Compound VII was given by all four
dodecafluorides, and further dehydrofluorination afforded both dienes (IV
and V). Compound VIII (gc retention time less than that of VII) was given
only by IIIt and IIIc, and on further dehydrofluorination gave only the
1,4-diene(IV). Clearly, therefore, all three were undecafluorocyclohept-
1-enes, compound VI the 3H- ; VII the 4H- ; and VIII the 5H- isomer.

The structures of the parent compounds were established by these
studies as 1H,3H-dodecafluorocycloheptanes (IIt and IIc) and 1H,4H-dodeca-
fluorocycloheptanes (IIIt and IIIc). However, the rates of dehydrofluor-
ination differed very little among the four compounds,and gave no help in
deciding which were trans and which cis. Arguments presented earlier [1; 2;
3], based on the Auwers-Skita rule, suggested that the lower-boiling of
each pair,IIt and IIIt respectively, would be the trans-isomers. Some
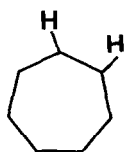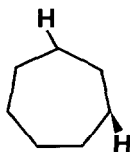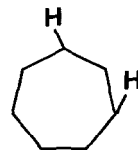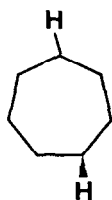further information was provided by ^{19}F nmr. Though the spectra were
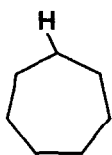
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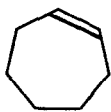
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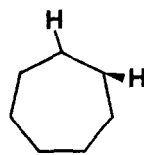
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II c

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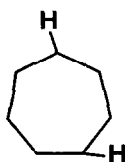
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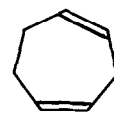
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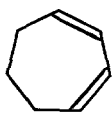
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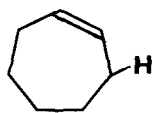
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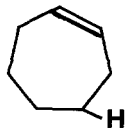
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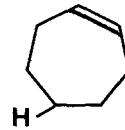
VIII

All unmarked bonds are to FLUORINE

SCHEME

complex, that from IIt had a broad singlet at 113.3 p.p.m., attributable
to the CF_2 group at position 2; however, in that from IIc, the bands from
the 2 position were much more complex. Compound IIIt showed a broad singlet
at 126.0 p.p.m., attributable to position 6, and surrounded by two AB
quartet systems, from positions 2,3,5 and 7. In contrast, IIIc gave peaks
in this general region analysable as 3 AB quartet systems, in intensity
ratio 2:2:1. From the symmetry, in IIt the fluorines on C_2 are equivalent
(not so in IIc), as are those on C_6 in IIIt (not for IIIc) [cf. 4].

The structural allocations for all these compounds therefore, in order
of increasing boiling points and glc retention times, are 1H/3H-(IIt);
1H/4H-(IIIt); 1H,3H/-(IIc); 1H,4H/-dodecafluorocycloheptane(IIIc). IIt
and IIIt were major products, IIIt slightly in excess, whilst smaller
amounts of IIc and IIIc were present, as would be expected [cf. 3].

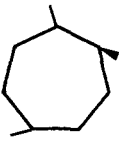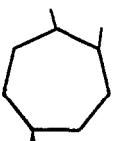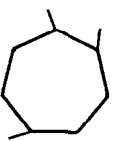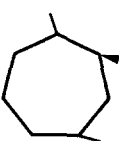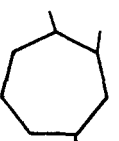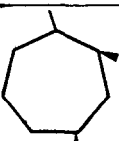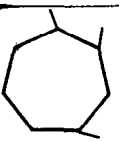It was decided to confirm these allocations by independent syntheses
based on chlorine addition to olefins VII and VIII, followed by lithium
aluminium hydride reduction to give 1H,2H,4H- and 1H,2H,5H-undecafluoro-
cycloheptanes. Mild fluorinations of these would then give specific
dodecafluorocycloheptanes in particular combinations, again following
procedures established in the cyclohexane [2] and cyclopentane series [3].
Since unsaturated by-products were formed in the reduction of the dichloro-
adduct of mono-ene D by lithium aluminium hydride at 0° [1], the present
reductions were done at -78°.

Both series of reactions proceeded smoothly. The simpler 1,2,5-
system was done first, olefin VIII giving a mixed dichloro-adduct (IX),
which reacted smoothly with lithium aluminium hydride in diethyl ether at
-78°. The product was separable by glc into three stereoisomeric 1H,2H,5H-
undecafluorocycloheptanes (Xa; Xb; Xc): there should be three such
compounds. A similar sequence was carried out from olefin VII, through a
dichloride (XI), to give four stereoisomeric 1H,2H,4H-undecafluorocyclo-
heptanes (XIIa; XIIb; XIIc; XIId); again in accord with the expected
number of products.

The Table summarises the results of individual fluorinations of each
of the seven undecafluorides by cobalt(III) fluoride in a small reactor at
160°. There were a few gaps in the results, both in analytical data and
isolation of compounds, due to lack of material in certain places.
Nevertheless, taken as a whole, the sequences gave a convincing and
unequivocal confirmation of the structural allocations of the dodeca-
fluorides (IIt; IIc; IIIt; IIIc), and enabled the seven undecafluorides

TABLE

Fluorinations of Undecafluorocycloheptanes by Co(III)F$_3$

Starting Material	Dodecafluorocycloheptanes Formed					
	1H,2H		1H,3H		1H,4H	
	trans	cis	trans	cis	trans	cis
	It	Ic	IIt	IIc	IIIt	IIIc
Xa	glc ir	–	–	–	glc ir	glc ir
Xb	–	M	–	–	glc ir	–
Xc	–	glc	–	–	–	glc ir
XIIa	glc ir	–	glc ir	–	–	glc ir
XIIb	–	M	glc ir	–	glc ir	–
XIIc	glc	–	–	glc	glc ir	–
XIId	–	glc	–	glc ir	–	glc ir

Key to TABLE

\	Hydrogen substituent on same side of ring plane
❯	Hydrogen substituent on opposite side of ring plane
glc	identity established by glc retention times on at least two and where possible three different packings
ir	identity established by infrared
M	expected product not detected

(Xa; Xb; Xc; XIIa; XIIb; XIIc; XIId) to be completely characterised (See Table for structures and Experimental for names). It is noteworthy that the highest boiling compound from each series (Xc and XIId) emerged as having the all-cis arrangement, as would be expected [2,3].

The two undecafluorides of lowest boiling points (Xa; XIIa) were shown by glc and ir to be present in fraction 7 of the original fluorination mixture. Others were almost certainly also present in the higher-boiling residues, but these were not separated in this work.

EXPERIMENTAL

Gas liquid chromatography - Preparative work was done in copper tubes 4.8 m long as follows: Tube A; 35 mm diam.; dinonyl phthalate/celite (1:2): Tube B; 75 mm diam.; dinonyl phthalate/celite (1:5): Tube C; 35 mm diam.; silicone gum/celite (1:5). Semi-preparative work was done in tubes 2 m long x 15 mm diam. Tube D was packed with dicyanodiethyl ether/celite 1:9, and Tube E with dinonyl phthalate/celite (1:2).

Spectroscopy. Nmr spectra [5] were measured in solutions in carbon tetrachloride on a Varian HA 100 instrument, ^{1}H at 100 MHz from tetramethyl silane as internal reference (τ units) and ^{19}F at 94.1 MHz from trichloro-fluoromethane as internal reference (δ units, negative), or on a Perkin Elmer R10 instrument (^{1}H at 60 MHz; ^{19}F at 56.4 MHz). Results are expressed as chemical shift position and type of signal, relative intensity (in brackets), ring position.

Infrared spectra (ir) were measured as liquid films on Perkin Elmer 257 or Infracord spectrophotometers.

Fluorination of cycloheptane. Cycloheptane (150 g) was passed during 3 h through a reactor at 180-190°, containing CoF_3 (10 Kg). The reactor was

then swept with nitrogen (20 ℓ h^{-1}) for 2 h. After collection in a trap at
−78°, being washed well with water and dried, the product (ca 300 g) showed
9 major peaks on analytical glc. All peaks were complex. Distillation
(2513 g), through a 4' vacuum-jacketed column packed with Dixon gauzes,
gave four fractions similar to those obtained before [1] in which the first
four peaks, respectively, were concentrated.

Fraction 1, b.r. <81.5° (222 g), and fraction 2, b.r. 81.5-89° (212 g),
corresponded to those described before [1], except that fraction 2
contained compound T in greater proportion.

Fraction 3, b.r. 89-92° (205 g) was fairly pure tridecafluorocyclo-
heptane (T) [1], obtained pure by glc (B, 75°, 50). An intermediate
fraction, b.r. 92-95° (121 g) consisted of T, and the components of
fraction 4, in roughly equal proportions.

Fraction 4, b.r. 96-100° (204 g) was separated by glc (B, 80°, 60;
8.2 g passed) to give:- (i) T (trace): (ii) a dodecafluoromethylcyclo-
hexane (1.1 g): (iii) 1H/2H-dodecafluorocycloheptane(It) (0.8 g),
identified by glc and ir [1]: (iv) a dodecafluoromethylcyclohexane (2.6 g):
(v) compounds IIt + IIIt (1.3 g). Sub fractions 4(ii) and 4(iv) analysed
as $C_7H_2F_{12}$, and trifluoromethyl groups were shown by ^{19}F nmr. Each was
mixed isomers of trifluoromethylnonafluorocyclohexane [cf. 6].

An intermediate fraction, b.r. 100-101.5° (162 g) contained the
components of fraction 4, together with those of 5, which predominated.

Fraction 5 (480 g) had b.r. 101.5-108°, and had two major components
in roughly equal amounts. Preparative glc (B, 100°, 60) on 10 g portions
gave:- (i) 1H/3H-dodecafluorocycloheptane(IIt) nc, (3.7 g) b.p. 105-106°
(Found: C, 26.7; H, 0.6. $C_7H_2F_{12}$ requires C, 26.8; H, 0.6%); nmr, ^{1}H,
4.89, bd, J=44; ^{19}F, 113.3 bs (1) 2; 118.8 and 122.6, J=291, 123.0 and
130.7, J=289, 2 AB quartets (4) 4, 5, 6, and 7; 220.0, bd, J=44 (1) 1 and
3:

(ii) 1H/4H-dodecafluorocycloheptane(IIIt) nc, (4.5 g) b.p. 107-108°
(Found: C, 27.2; H, 0.7%); nmr, ^{1}H, 4.89, bd, J=44; ^{19}F, 115.8 and
125.5, J=289, 121.2 and 124.0, J=294, 2 AB quartets (4) 2, 3, 5 and 7;
126.0 , bs (1) 6; 224.0, bd, J=44, (1) 1 and 4. Compounds IIt and IIIt
each contained <5% impurity (−CHF$_2$ groups shown by ^{19}F nmr).

Fraction 6 (115 g), b.r. 108-114°, was complex. It gave on glc
(B, 100°, 65; 9.5 g separated):- (i) IIt, (0.7 g): (ii) IIIt, (1.3 g):
(iii) a mixture (4.9 g) (Found: C, 26.9; H, 0.5%), containing two new
compounds, the shorter-retained in roughly 2:1 excess, and minor constituents.

Further separation of sub-fraction 6(iii) by glc (D, 45°, 3) each fraction further separated twice gave:- 6(iii)a,1H,3H/-dodecafluorocyclo-heptane(IIc) nc, b.p. 112-113°; nmr, ^{1}H, 4.95, bd, J=44; ^{19}F, 112-133 complex series of peaks (5) 2,4,5,6 and 7; 217.0, bd, J=45 (1) 1 and 3: 6(iii)b, 1H,4H/-dodecafluorocycloheptane(IIIc) nc, b.p. 113-114°; nmr, ^{1}H, 4.98, bd, J=45; ^{19}F, 113-130 complex series of peaks probably 3 AB quartets (2:2:1) 2,3,5,6 and 7; 219.0, bd, J=45, (1) 1 and 4. Compound IIc had 5-10% impurity present ($-CHF_2$ group by ^{19}F nmr).

Fraction 7 (103 g) b.r. 114-117° gave on glc (B, 100°, 65; 10 g passed):- (i) IIc + IIIc (1.1 g): (ii) a mixture (4.9 g), shown by glc using various packings and ir to be mainly Xa and XIIa (see later).

The residue, b.r. >117° (676 g) was not examined.

Analytical glc on several packings, and ir, confirmed the earlier result [1], that 1H,2H/-dodecafluorocycloheptane(Ic) was not detectable in the fluorination mixture, and could have been present in only a very small amount.

Dehydrofluorination of the trans-1H/3H-dodecafluoride(IIt). IIt (10.0 g) potassium hydroxide (10 g) and water (30 cm^3) were refluxed together for 8 h. The organic layer (7.9 g) was separated off and dried and gave by glc (A, 65°, 15):- (i) decafluorocyclohepta-1,4-diene(IV) nc (0.6 g), b.p. 85-86° (Found: C, 30.6. C_7F_{10} requires C, 30.7%); m/e 274 (M$^+$); ir 1750 (s) cm^{-1}; uv, no significant selective absorption above 200 nm; ^{19}F nmr, 103.2 (1) 3, 117.3 (2) 6 and 7, 147.3 (2) 1,2,4,5, all complex: (ii) deca-fluorocyclohepta-1,3-diene(V) nc (2.1 g), b.p. 88° (Found: C, 30.7; F, 69.2; m/e 274); ir 1725 (s); uv, λ_{max} 245 nm (ε 3425 in ethanol); ^{19}F nmr, 119.7 (2) 5 and 7, 128.8 (1) 6, 143.9 (1) 2 and 3, 146.8 (1) 1 and 4, all complex: (iii) 3H-undecafluorocyclohept-1-ene(VI) nc (0.4 g) (Found: C, 28.7; H, 0.1; F, 70.7. C_7HF_{11} requires C, 28.6; H, 0.3 F, 71.1%); ir 1750 (s), 3000 (w): (iv) 4H-undecafluorocyclohept-1-ene(VII) nc (1.0 g) (Found: C, 28.6; H, 0.3; F, 71.4%); ir 1730 (s) 2975 (w); nmr, ^{1}H, 4.87, bd, J=46; ^{19}F, 106.0 and 114.4, AB quartet, J=226 (4) 3 and 5, 118.7 c (2) 7, 128.5 c (2) 6, 141.4 c (1) 1, 144.2 c (1) 2, 207.3, bd, J=46 (1) 4: (v) unchanged IIt (2.1 g).

Oxidation of the 1,3-diene(V). Permanganate in acetone [7] afforded dianilinium hexafluoroglutarate (80%), m.p. 217-218°, with a correct ir spectrum.

Dehydrofluorination of the 3H-olefin(VI). Potassium hydroxide (3.0 g),
water (2.5 cm³) and VI (0.6 g) were refluxed together for 2 h. Analytical
glc showed only one (small) product peak, corresponding to diene V, and a
large peak due to unreacted VI. More dilute alkali gave only VI, whilst
molten potassium hydroxide caused almost complete decomposition.

Dehydrofluorination of the 4H-olefin(VII). VII (3.2 g) potassium hydroxide
(5.0 g) and water (12 cm³) were refluxed together for 5 h. The organic
layer gave by glc (A, 50°, 11):- (i) 1,4-diene(IV) (0.4 g): (ii) 1,3-
diene(V) (1.3 g): (iii) unreacted VII (0.2 g): all were identified by
glc and ir.

Dehydrofluorination of the trans-1H/4H-dodecafluoride(IIIt). Treatment as
for IIt gave product (8.3 g) separated by glc (A, 65°, 20) into:-
(i) 1,4-diene(IV) (2.1 g): (ii) 1,3-diene(V) (2.4 g): (iii) 5H-undeca-
fluorocyclohept-1-ene(VIII) nc, (1.2 g) (Found: C, 28.4; H, 0.4%), ir
1715 (s), 2980 and 3000 (w): (iv) 4H-ene(VII) (0.9 g): (v) unreacted
IIIt (0.4 g). Fractions (i), (ii), (iv), and (v) were identified by glc
and ir identity with authentic specimens.

Dehydrofluorination of the 5H-ene(VIII). VIII (6.0 g), potassium hydroxide
(6.0 g) and water (15 cm³) were refluxed together for 6 h. The organic
layer (4.1 g) by glc (A, 50°, 12) gave:- (i) 1,4-diene(IV) (1.8 g):
unreacted VIII (2.4 g): both were identified by glc and ir.

Dehydrofluorination of fraction 5. Fraction 5 above (mainly IIt + IIIt)
(70 g), potassium hydroxide (110 g), and water (220 cm³) were refluxed
together for 24 h, to give product (43.5 g). Glc (B, 50°, 45) gave the
major components:- (i) IV (9.3 g): (ii) V (19.5 g).

Dehydrofluorination of the 1H,3H/-dodecafluoride(IIc). IIc (1.0 g) was
treated as IIt for 7 h. Analytical glc on the product (0.6 g) gave a
trace with 5 peaks having retention times respectively identical to those
from the reaction on IIt. Samples of V, VII and IIc were isolated by glc
(E, 70°, 3) and identified by ir. Small comparative experiments showed
that the reaction rate was slightly greater than for IIt, and proportions
of products were roughly comparable, except that less olefin VI was present.

Dehydrofluorination of the 1H,4H/-dodecafluoride(IIIc). IIIc (1.5 g) was
refluxed with KOH (1.5 g) and water (3 cm³) for 4 h, and gave a product
(0.9 g), separated by glc (E, 60°, 3) into:- (i) IV: (ii) V, (iii) VIII
(iv) VII: (v) IIIc: all were identified by glc and ir. Small comparative

experiments showed that IIIt and IIIc reacted at similar rates to give products in comparable proportions.

1,2-Dichloro-5H-undecafluorocycloheptane isomers(IX). A stream of chlorine (dried by conc. H_2SO_4) was bubbled for 3.5 h through olefin VIII (12.0 g) in a flask irradiated by uv light. The product (IX) (14.7 g) (Found: C, 22.1; H, 0.4; Cl, 19.2. Calc. for $C_7HCl_2F_{11}$: C, 23.0; H, 0.3; Cl, 19.4%) showed, by glc, one major and two minor components, but no VIII.

1H,2H,5H-Undecafluorocycloheptanes(X). Mixture IX (13.0 g) in dry diethyl ether (25 cm^3) was added during 45 min to a stirred suspension of lithium aluminium hydride (2.26 g) in dry ether (130 cm^3) kept at -78°. The mixture was then allowed to stand, finally at 15° for 45 min. After being cooled to 0°, the mixture was cautiously treated with a slight excess of sulphuric acid (50% v/v), the ether layer separated, dried ($MgSO_4$), filtered, and the ether evaporated through a 6" fractionating column. Glc (C, 50°, 14) gave:- (i) diethyl ether: (ii) 1H,5H/2H-undecafluoro-cycloheptane(Xa) nc (3.4 g), b.p. 116-117°: (iii) 1H,2H/5H-undecafluoro-cycloheptane(Xb) nc (1.3 g), b.p. 129-130°: (iv) 1H,2H,5H/-undecafluoro-cycloheptane(Xc) nc (1.6 g), b.p. 140-141° (Found: C, 28.2; H, 0.9. $C_7H_3F_{11}$ requires C, 28.4; H, 1.0%).

Fluorinations of the 1H,2H,5H-undecafluorides(Xa - Xc). Each compound was passed in a stream of nitrogen (2 ℓ h^{-1}) over cobaltic fluoride at 160° in a small stirred reactor. The reactor was swept with nitrogen (4 ℓ h^{-1}) for 2 h further. The product (collected in a trap cooled by liquid nitrogen) was washed with water, dried, and identified by analytical glc. Columns were used with packings of dinonyl phthalate/celite 1:10 (6 m tube, 100°), Kel F oil/celite 1:5, and dicyanodiethyl ether/celite 1:9, (both 2 m tubes, 52-70°), N_2 flow rates 1.5 ℓ h^{-1}. In all cases, identities were established by retention times over at least 2 and, wherever possible, 3 packings. In some cases (marked ir) identities could be confirmed by infrared spectra, on small samples isolated using the columns semi-preparatively.

Compound Xa (1.5 g) gave 1H/2H- (ir) (It) [1]; 1H/4H (ir) (IIIt); and 1H,4H/- (ir) (IIIc)-dodecafluorocycloheptane, together with C_7F_{14}, T (ir), and Xa (ir).

Compound Xb (0.9 g) gave the 1H/4H-dodecafluoride (ir) (IIIt); together with T, and Xb (ir).

Compound Xc (1.0 g) gave the 1H,2H/- (Ic) [1], and 1H,4H/- (ir) (IIIc)-dodecafluorides, together with C_7F_{14}, T (ir), and Xc.

1H,2H,4H-Undecafluorocycloheptanes(XII). Olefin VII (10.0 g) was treated
with chlorine as for VIII to give a crude dichloro-adduct (XI) (11.5 g).
Treatment of this by lithium aluminium hydride as for IX gave, on glc
separation (A, 100°, 15):- (i) ether: (ii) 1H,4H/2H-undecafluorocyclo-
heptane(XIIa) nc (2.1 g), b.p. 117-118°: (iii) the 1H,2H/4H-isomer nc
(XIIb) (1.4 g), b.p. 120-121° (Found: C, 28.6; H, 1.1%): (iv) the
1H/2H,4H-isomer(XIIc) nc (1.8 g), b.p. 121-122° (Found: C, 28.4; H,
0.8%): (v) the 1H,2H,4H/-isomer(XIId) nc (1.9 g), b.p. 128-129°
(Found: C, 28.5; H, 1.0%).

Fluorinations of the 1H,2H,4H-Undecafluorides(XIIa - XIId). Fluorinations
and identifications were as for Xa - c . XIIa (1.5 g) gave 1H/2H- (ir)
(It); 1H/3H- (ir) (IIt); and 1H,4H/- (ir) (IIIc)-dodecafluorides,
together with T (ir), and XIIa (ir). XIIb (1.0 g) gave 1H/3H- (ir) (IIt);
and 1H/4H- (ir) (IIIt)-dodecafluorides; and XIIb (ir). XIIc (1.1 g) gave
1H/2H- (It); 1H,3H/- (IIc); and 1H/4H- (ir) (IIIt)-dodecafluorides, with
T, and XIIc (ir). XIId (1.4 g) gave 1H,2H/- (Ic); 1H,3H/- (ir) (IIc);
and 1H,4H/- (ir) (IIIc)-dodecafluorides; T (ir); and XIId.

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