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Polyfluoro-triphenylenes derived from fluoroaromatic lithium reagents; the crystal and molecular structure of hexadecafluoro-1-phenyltriphenylene

C.A. Beaumont, J. Bowen Jones, D.S. Brown, A.G. Massey and J. Watkin

Department of Chemistry, University of Technology, Loughborough, Leics., LE11 3TU (Great Britain)

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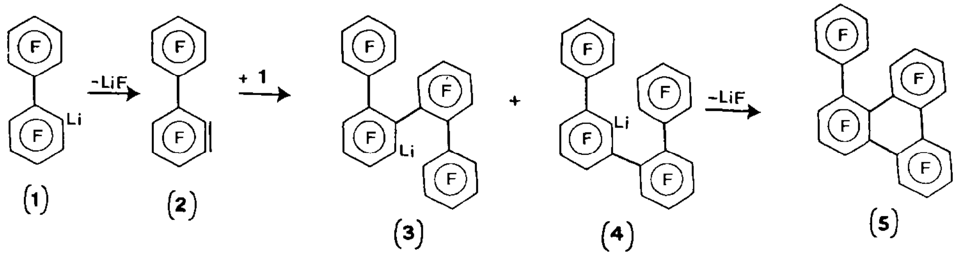
Abstract

The products formed upon addition of 2-lithio-2′-halobiphenyls to perfluoroarynes yield polyfluoro-triphenylenes via an intramolecular loss of lithium halide. Thus hexadecafluoro-1-phenyltriphenylene was obtained by thermal decomposition of 2-nonafluorobiphenyllithium; its structure has been confirmed by X-ray crystallography. Similar reactions gave 1,2,3,4-tetrafluorotriphenylene and 1,2,3,4,5,6,7,8-octafluorotriphenylene.

Introduction and discussion

The thermal decomposition of 2-nonafluorobiphenyllithium (**1**) is known to proceed via the perfluoroaryne **2**, which may be trapped with either furan or benzene [1]. We have now shown that in the absence of such trapping agents, **2** adds 2-nonafluorobiphenyllithium to give the quaterphenyllithium reagents **3** and **4** which cyclise via loss of lithium fluoride to form hexadecafluoro-1-phenyltriphenylene (**5**).



As sometimes happens with highly fluorinated organic species, problems were encountered with the fluorine analyses for **5** but the molecular formula was confirmed as $C_{24}F_{16}$ by mass spectrometry. The presence of 14 magnetically different fluorine atoms resulted in considerable overlap of peaks in the ^{19}F NMR

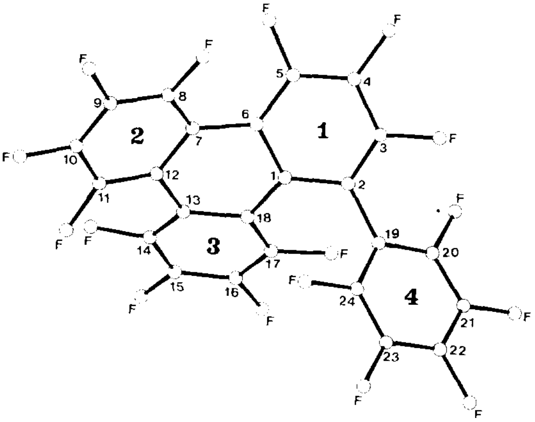
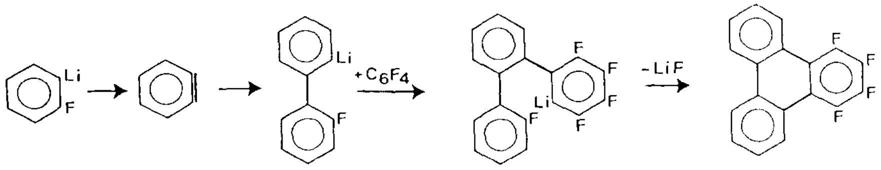


Fig. 1. ORTEP drawing of $C_{24}F_{16}$ showing ring and atom numbering.

spectrum; but the structure of **5** was strongly supported by the presence of a triplet due to F(4) (see Fig. 1) at 149.6 ppm (relative to $CFCl_3$). A group of several peaks between 121 and 125 ppm probably arises from the various "*ortho*" fluorines on the triphenylene rings, whilst the "*meta*" fluorine (and F(22)) peaks all fall in the narrow range 145.5–147 ppm; fluorines F(20),F(24) and F(21),F(23) of the C_6F_5 ring give rise to peaks of relative intensity two at 132.6 and 155.8 ppm, respectively. Confirmation of the structure was accomplished by X-ray crystallography, as described later. In contrast to dodecafluorotriphenylene and triphenylene, which form a crystalline 1 : 1 "complex" virtually instantaneously, **5** and triphenylene were found not to react together even on slow crystallization during some weeks. The adverse steric effect of the pentafluorophenyl group on **5** presumably prevents efficient packing of the two components in the solid phase.

This two-stage synthesis of **5** starting from the relatively cheap bromopentafluorobenzene represents the most convenient available route to a perfluorotriphenylene. Evidence to support the proposed mechanism comes from the presence of a peak at $m/e = 612$ ($C_{24}F_{17}H^+$) in the mass spectra of impure samples of **5**; this is probably the parent ion of the hydrolysis products arising from traces of uncyclised lithium reagents **3** and **4** remaining until reaction work-up.

Previous research in these laboratories, reported by Heaney and his co-workers [2], showed that triphenylenes result from the formation of arynes in the presence of preformed biphenyl derivatives. It is possible in some cases, however, to take advantage of the exceedingly rapid low-temperature production of 2-lithio-2′-halobiphenyls from 2-lithiohalobenzenes to form the biphenyls in situ. For example, if two moles of butyllithium are added to one mole each of 2-iodofluorobenzene and bromopentafluorobenzene, the major product is 1,2,3,4-tetrafluorotriphenylene (**6**):



(**6**)

Unexpectedly, when two moles of butyllithium were added to one mole each of 2-iodofluorobenzene and 2-bromononafluorobiphenyl large amounts of "$C_{12}F_9$-$C_{12}H_9$" were isolated; this may arise, via hydrolysis during work-up, from the two lithium reagents **7** and **8** which had failed to cyclise. A small quantity of 1,2,3,4,5,6,7,8-octafluorotriphenylene was also isolated from among the products, the low yield presumably reflecting the relative instability of 2-lithiofluorobenzene (most of which would rapidly form 2-lithio-2′-fluorobiphenyl under the reaction conditions).

Table 1

Atomic coordinates ($\times 10^4$) with esd's

Atom	x/a	y/b	z/c	B_{eq} (Å^2) ^{a}
C(1)	3306(3)	9063(2)	11099(4)	3.0(2)
C(2)	4395(3)	8800(2)	10901(5)	3.4(2)
C(3)	4645(3)	8784(2)	9481(5)	4.0(2)
C(4)	3877(4)	8985(2)	8241(5)	4.3(2)
C(5)	2909(4)	9330(2)	8469(5)	3.8(2)
C(6)	2648(3)	9448(2)	9890(4)	3.1(2)
C(7)	1836(3)	9978(2)	10276(5)	3.1(2)
C(8)	1634(3)	10613(2)	9449(5)	3.6(2)
C(9)	983(4)	11154(2)	9892(5)	3.9(2)
C(10)	588(4)	11107(2)	11207(6)	4.1(2)
C(11)	840(3)	10510(2)	12121(5)	3.6(2)
C(12)	1433(3)	9932(2)	11652(5)	3.1(2)
C(13)	1714(3)	9284(2)	12570(4)	3.0(2)
C(14)	1012(3)	9009(2)	13507(5)	3.6(2)
C(15)	1275(4)	8399(2)	14352(5)	4.2(2)
C(16)	2238(4)	8034(2)	14187(5)	4.0(2)
C(17)	2923(3)	8264(2)	13210(5)	3.4(2)
C(18)	2715(3)	8898(2)	12369(4)	3.0(2)
C(19)	5308(3)	8629(2)	12183(5)	3.3(2)
C(20)	5938(3)	8001(2)	12240(5)	3.8(2)
C(21)	6851(3)	7875(2)	13348(5)	4.1(2)
C(22)	7131(3)	8374(3)	14448(5)	4.4(2)
C(23)	6513(4)	8987(3)	14464(5)	4.5(2)
C(24)	5619(3)	9112(2)	13327(5)	3.8(2)
F(3)	5679(2)	8566(1)	9225(3)	5.4(1)
F(4)	4112(2)	8873(1)	6852(3)	5.8(2)
F(5)	2181(2)	9529(1)	7240(3)	5.1(1)
F(8)	2140(2)	10741(1)	8250(3)	4.9(1)
F(9)	797(2)	11751(1)	9052(3)	5.5(1)
F(10)	−6(2)	11646(1)	11693(3)	5.9(2)
F(11)	555(2)	10536(1)	13485(3)	5.1(1)
F(14)	−11(2)	9300(1)	13571(3)	4.7(1)
F(15)	576(2)	8150(1)	15243(3)	6.1(2)
F(16)	2472(2)	7414(1)	14922(3)	5.8(2)
F(17)	3759(2)	7809(1)	12984(3)	4.7(1)
F(20)	5647(2)	7490(1)	11210(3)	5.0(1)
F(21)	7436(2)	7263(1)	13364(3)	5.7(2)
F(22)	8008(2)	8244(2)	15552(3)	6.2(2)
F(23)	6788(2)	9478(2)	15546(3)	6.6(2)
F(24)	5049(2)	9733(1)	13332(3)	5.1(1)

^{a} $B_{eq} = \frac{1}{3}\Sigma_i B_{ii}$.

Table 2

Bond lengths (Å) and bond angles (°) in hexadecafluoro-1-phenyltriphenylene with e.s.d.'s in parenthesis

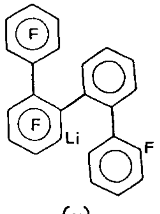
C(1)–C(2)	1.425(5)	C(14)–C(15)	1.377(6)
C(2)–C(3)	1.360(6)	C(15)–C(16)	1.363(6)
C(3)–C(4)	1.376(6)	C(16)–C(17)	1.363(6)
C(4)–C(5)	1.366(6)	C(17)–C(18)	1.406(5)
C(5)–C(6)	1.382(6)	C(13)–C(18)	1.428(5)
C(6)–C(1)	1.428(5)	C(18)–C(1)	1.470(5)
C(6)–C(7)	1.465(5)	C(2)–C(19)	1.487(5)
C(7)–C(8)	1.400(5)	C(19)–C(20)	1.387(5)
C(7)–C(12)	1.403(6)	C(20)–C(21)	1.371(6)
C(8)–C(9)	1.372(6)	C(21)–C(22)	1.361(6)
C(9)–C(10)	1.347(6)	C(22)–C(23)	1.362(6)
C(10)–C(11)	1.389(6)	C(23)–C(24)	1.372(6)
C(11)–C(12)	1.392(5)	C(24)–C(19)	1.375(6)
C(12)–C(13)	1.472(5)		
C(13)–C(14)	1.380(5)		

C(3)–F(3)	1.352(4)	C(15)–F(15)	1.330(4)
C(4)–F(4)	1.343(5)	C(16)–F(16)	1.340(4)
C(5)–F(5)	1.342(5)	C(17)–F(17)	1.349(4)
C(8)–F(8)	1.340(5)	C(20)–F(20)	1.338(4)
C(9)–F(9)	1.344(5)	C(21)–F(21)	1.336(5)
C(10)–F(10)	1.344(5)	C(22)–F(22)	1.342(5)
C(11)–F(11)	1.328(5)	C(23)–F(23)	1.339(5)
C(14)–F(14)	1.343(4)	C(24)–F(24)	1.343(4)

C(2)–C(1)–C(6)	118.4(4)	C(11)–C(12)–C(13)	123.2(4)
C(2)–C(1)–C(18)	125.8(4)	C(13)–C(12)–C(7)	117.9(4)
C(6)–C(1)–C(18)	115.3(3)	C(12)–C(13)–C(18)	117.5(4)
C(1)–C(2)–C(3)	117.9(4)	C(12)–C(13)–C(14)	122.9(4)
C(1)–C(2)–C(19)	122.9(4)	C(14)–C(13)–C(18)	119.4(3)
C(3)–C(2)–C(19)	118.8(4)	C(13)–C(14)–C(15)	122.6(4)
C(2)–C(3)–C(4)	122.6(4)	C(13)–C(14)–F(14)	121.3(4)
C(2)–C(3)–F(3)	120.8(4)	C(15)–C(14)–F(14)	115.9(4)
C(4)–C(3)–F(3)	116.6(4)	C(14)–C(15)–C(16)	118.3(4)
C(3)–C(4)–C(5)	118.3(4)	C(14)–C(15)–F(15)	120.9(4)
C(3)–C(4)–F(4)	120.2(4)	C(16)–C(15)–F(15)	120.7(4)
C(5)–C(4)–F(4)	121.5(4)	C(15)–C(16)–C(17)	121.0(4)
C(4)–C(5)–C(6)	122.4(4)	C(15)–C(16)–F(16)	119.3(4)
C(4)–C(5)–F(5)	117.0(4)	C(17)–C(16)–F(16)	119.5(4)
C(6)–C(5)–F(5)	120.6(4)	C(16)–C(17)–C(18)	122.8(4)
C(1)–C(6)–C(5)	116.7(4)	C(16)–C(17)–F(17)	115.7(3)
C(1)–C(6)–C(7)	117.4(4)	C(18)–C(17)–F(17)	121.3(4)
C(5)–C(6)–C(7)	125.6(4)	C(17)–C(18)–C(1)	122.1(3)
C(6)–C(7)–C(8)	120.6(4)	C(17)–C(18)–C(13)	115.7(4)
C(6)–C(7)–C(12)	120.1(3)	C(1)–C(18)–C(13)	120.5(3)
C(8)–C(7)–C(12)	118.0(4)	C(2)–C(19)–C(20)	122.3(4)
C(7)–C(8)–C(9)	121.6(4)	C(2)–C(19)–C(24)	121.0(4)
C(7)–C(8)–F(8)	121.3(4)	C(20)–C(19)–C(24)	116.6(4)
C(9)–C(8)–F(8)	116.9(4)	C(19)–C(20)–C(21)	122.2(4)
C(8)–C(9)–C(10)	120.2(4)	C(19)–C(20)–F(20)	119.9(4)
C(8)–C(9)–F(9)	119.7(4)	C(21)–C(20)–F(20)	117.9(4)
C(10)–C(9)–F(9)	120.0(4)	C(20)–C(21)–C(22)	118.9(4)
C(9)–C(10)–C(11)	120.1(4)	C(20)–C(21)–F(21)	120.3(4)
C(9)–C(10)–F(10)	121.0(4)	C(22)–C(21)–F(21)	120.7(4)
C(11)–C(10)–F(10)	118.9(5)	C(21)–C(22)–C(23)	120.9(4)
C(10)–C(11)–C(12)	120.9(4)	C(21)–C(22)–F(22)	119.1(4)

Table 2 (continued)

C(10)–C(11)–F(11)	117.1(4)	C(23)–C(22)–F(22)	120.0(4)
C(12)–C(11)–F(11)	121.8(4)	C(22)–C(23)–C(24)	119.4(4)
C(7)–C(12)–C(11)	118.9(4)	C(22)–C(23)–F(23)	120.9(4)
C(24)–C(23)–F(23)	119.7(4)		
C(23)–C(24)–C(19)	122.0(4)		
C(23)–C(24)–F(24)	118.4(4)		
C(19)–C(24)–F(24)	119.6(4)		



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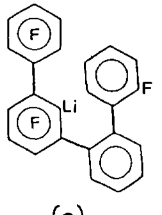


(8)

In view of the poor fluorine analyses and the rather tenuous evidence provided by its crowded ^{19}F NMR spectrum, the structure of **5** was confirmed using X-ray crystallography; an ORTEP diagram, the atom numbering and the ring numbering are given in Fig. 1. Tables 1 and 2 list the atomic coordinates and molecular dimensions, respectively. It should be noted that the atom numbering in Fig. 1 places C(1) in the central ring, and so the numbers are not related to those used in substitution nomenclature.

The triphenylene ring system is severely distorted owing to steric interaction between the "*ortho*" fluorines and this makes the synthesis of **5** that much more remarkable. Even with the molecular distortion the "*ortho*" fluorines are forced closer together than would be expected from the Van der Waals radius for fluorine of 1.35 Å: F(5) – F(8) 2.442(7), F(11) – F(14) 2.408(6) Å. The effects of the strain are manifested by ring buckling, particularly in ring 1 (see Fig. 1), and by deviation of many angles from the ideal value of 120°. As an example of the latter effect, the F(5) – F(8) congestion closes angles C(4)C(5)F(5) and C(9)C(8)F(8) to 117.0(4) and 116.9(4)°, respectively, but opens up angle C(5)C(6)C(7) to 125.6(4)°. In crystalline dodecafluorotriphenylene, $C_{18}F_{12}$, the molecule had a C_2 axis passing through one of the rings [1]. In **5**, ring 1 approximately possesses a C_2 axis and the molecule as a whole is distorted in the same sense as $C_{18}F_{12}$. See Table 3 for dihedral angles.

There also appears to be considerable strain between ring 4 and F(17): the angle C(16)C(17)F(17) is reduced to 115.7(3)°, the angle C(1)C(2)C(19) is opened up to

Table 3

Dihedral angles (°) between the rings with esd's [a]

ring(1)–ring(2)	39(7)
ring(1)–ring(3)	42(7)
ring(1)–ring(4)	50(7)
ring(2)–ring(3)	28(3)

[a] The esd's are high due to the very distorted nature of rings 1, 2 and 3.

122.9(4)°, and the bond between C(1) and C(2) is significantly longer than bonds in comparable ring positions in other parts of the molecule; however, C(19) and F(17) are still only 2.59(5) Å apart. The pentafluorophenyl group, on the other hand, suffers relatively little distortion.

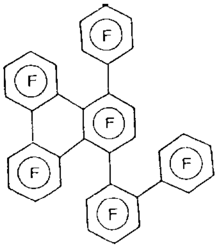
Experimental

All reactions involving lithium reagents were carried out under dry nitrogen and employed 0.01 mole quantities of reagents. The preparation of 2-nonafluorobiphenyllithium has been described previously [1].

Decomposition of 2-nonafluorobiphenyllithium

Butyllithium (1.6 *M* in hexane) was added at −90 °C to an equimolar amount of 2-bromononafluorbiphenyl dissolved in diethyl ether and the mixture stirred for 15 min. The mixture was then allowed to warm up slowly to room temperature and stirring continued overnight. Analytical TLC showed the presence of one major and four minor components, the former giving the fourth band from the solvent front. Preparative TLC on alumina allowed separation of all the products, band 4 being shown to be hexadecafluoro-1-phenyltriphenylene (**5**), m.p. 133–134 °C (Found for two different samples: C, 48.65, 48.4; H, 0.1, 0.0; F, 49.5, 49.4%; $C_{24}F_{16}$ calc: C, 48.7, H, 0.0; F, 51.3%). The molecular ion gave rise to the base peak in the mass spectrum: measured mass 591.975 (calcd. for $^{12}C_{24}{}^{19}F_{16}{}^{+}$, 591.974); 592.980 ($^{12}C_{23}{}^{13}C^{19}F_{16}{}^{+}$, 592.978); 593.980 ($^{12}C_{22}{}^{13}C_{2}{}^{19}F_{16}{}^{+}$, 593.981); the major fragmentation peaks were at 573 ($P - F$; 12%), 554 ($P - 2F$; 14%), 542 ($P - CF_3$; 39%), 523 ($P - C_2F_3$; 79%) and 492 ($P - C_2F_4$; 44%). A cross-scan report showed a close correlation between the parent ion, the above fragment ions and the total ion current; the doubly-charged parent ion was also present with a relative intensity of 11%.

The next most-major reaction product gave rise to the slowest moving band (band 5) on the TLC plate; the molecular ion appeared at 888 m.u. in the mass spectrum: measured masses for the singly- and doubly-charged molecular ions were 887.966 (calcd. for $^{12}C_{36}{}^{19}F_{24}{}^{+}$, 887.962) and 443.980 ($^{12}C_{30}{}^{19}F_{24}{}^{2+}$, 443.980). The infrared spectrum was very similar to that of **5**, but with bands typical of a $C_{12}F_9$ fragment superimposed on it. We assume this compound is one, or both, of the isomers **9** and **10** arising via aryne formation from lithium reagent **3** followed by addition of 2-nonafluorobiphenyllithium and subsequent cyclisation. The material in band 3 had $C_{24}F_{16}Br^{+}$ as its molecular ion in the mass spectrum and probably arises, as two isomers, from Li/Br exchange between unused 2-Br$C_{12}F_9$ and lithium reagents **3** and **4**.



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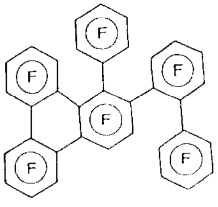

(10)

Simultaneous decomposition of 2-lithiofluorobenzene and pentafluorophenyllithium

To a stirred, ethereal solution of equimolar amounts of 2-iodofluorobenzene and bromopentafluorobenzene held at −90 °C was added two moles of butyllithium (1.6 *M* in hexane). After 15 min the mixture was allowed to warm to room temperature and stirring continued overnight. The major product isolated by preparative TLC on alumina was shown to be 1,2,3,4-tetrafluorotriphenylene, m.p. 229 °C, by comparison of its infrared spectrum with that of an authentic sample [2]. By far the most intense peak in the mass spectrum was the parent ion: measured mass 300.055 (calcd. for $^{12}C_{18}{}^{18}F_4{}^{1}H_8{}^{+}$, 300.056) and 301.058 ($^{12}C_{17}{}^{13}C^{19}F_4{}^{1}H_8{}^{+}$, 301.059); a strong doubly-charged parent ion, at 45% relative intensity was also present: measured mass 150.028 (calcd. for $^{12}C_{18}{}^{19}F_4{}^{1}H_8{}^{2+}$, 150.028) and 150.529 ($^{12}C_{17}{}^{13}C^{19}F_4{}^{1}H_8{}^{2+}$, 150.529). Doubly-charged ions, corresponding to the fragments $P-\mathrm{H}$, $P-2\mathrm{H}$, $P-\mathrm{CF_2}$ and $P-26$ gave peaks with intensities between 5 and 13%. Four very minor components, detectable by TLC, were not studied.

When this experiment was repeated but starting from 2 mol of C_6F_5Br, 1 mol 2-IFC_6H_4 and 3 mol of butyllithium it was found that the major product was precipitated on addition of 40–60 °C petroleum ether to an ether solution. This material analysed as "$C_{12}F_9C_{12}H_9$" and may be the hydrolysis products of lithium reagents **7** and **8** [Found: C, 61.3; H, 1.9; F, 36.7; $C_{24}H_9F_9$ calc: C, 61.5; H, 1.9, F, 36.5%]. The reason for the non-cyclisation of **7** and **8** via lithium fluoride elimination is not clear.

Simultaneous decomposition of 2-lithiofluorobenzene and 2-nonafluorobiphenyllithium

To 1 mol each of 2-bromononafluorobiphenyl and 2-iodofluorobenzene in diethyl ether were added 2 mol of butyllithium at −90 °C. The stirred mixture was held at −90 °C for 15 min then allowed to warm slowly to room temperature and stand overnight. The solution was filtered to remove lithium fluoride and the ether solvent removed under reduced pressure to leave a thick brown oil. Heating this oil in a sublimer tube held at 100 °C (10^{-3} mm Hg) gave a small quantity ($<5\%$) of 1,2,3,4,5,6,7,8-octafluorotriphenylene as colourless, needle crystals, m.p. 140 °C [Found: C, 58.4; H, 1.5; $C_{18}H_4F_8$ calc: C, 58.1; H, 1.1%. The parent ion gave rise to the base peak in the mass spectrum and no other peaks having an intensity greater than 10% were present: measured mass 372.011 (calcd. for $^{12}C_{18}H_4{}^{19}F_8$, 372.018) and 373.016 ($^{12}C_{17}{}^{13}C\ H_4{}^{19}F_8$, 373.022).

Determination of the structure of $C_{24}F_{16}$

Crystal data: $C_{24}F_{16}$, $M_r = 592.238$, monoclinic, $P2_1/c$, *a* 11.907(5), *b* 18.664(4), *c* 9.008(9) Å, *β* 99.33(4)°, *U* 1975.39 Å³, $Z = 4$, D_m (flotation) 1.986, D_x 1.992 g cm^{-3}, λ(Mo-K_α) 0.71069 Å, *μ* 1.62 cm^{-1}, $F(000) = 1152$, *T* 293 K. Final $R = 0.039$ for 1955 observed reflections with $I > 3\sigma(I)$.

A single crystal, 0.5 × 0.3 × 0.1 mm, was sealed in a Lindemann-glass capillary, mounted about *c*. Preliminary lattice constants were determined from oscillation and Weissenberg photographs; refined lattice constants from a Stoe Stadi-2 two-circle diffractometer, using axial-row reflections with a *θ* range of 6 to 20°. Corrections were applied for Lp, but not for absorption and extinction. The intensities of 3353 reflections were measured out to a maximum 2*θ* of 60°, using Mo-K_α radiation; 1955 reflections had $I > 3\sigma(I)$ and were classed as observed. A standard check reflection was measured every 50 reflections for each layer, and no significant variation in intensity was noted. The structure was solved by direct methods using SHELX76 [3], implemented at the Loughborough University of Technology Computer Centre. Refinement by accelerated full-matrix least squares on F with unit weights, and allowing anisotropic motion for all atoms, gave $\Delta/\sigma < 0.002$, $\Delta\rho$ excursions = $+0.19$ to -0.23 e Å^{-3}, and a final $R = 0.039$. The scattering factors used were those of Cromer and Mann [4]. Geometry calculations using XRAY72 [5] were carried out at the University of Manchester Regional Computer Centre. A copy of the list of structure factors and anisotropic temperature factors is available, on request, from J.B.J.

Infrared spectra

$C_{24}F_{16}$ *(Nujol)*: 1651w, 1627w, 1599m, 1572wsh, 1520mb, 1496s, 1409m, 1358m, 1140vwb, 1099s, 1081s, 1066s, 1027m, 992s, 958m, 800w, 776m, 753vw, 735sh, 731m, 727m, 720s, 703w, 694m, 662w, 649m, 622w, 574vw, 346vw.

$C_{36}F_{24}$*(Nujol, band 5)*: 1653–1605bw, 1520sh, 1508sh, 1499s, 1410sh, 1347w, 1290wb, 1250wb, 1145sh, 1114m, 1082s, 1070s, 1058sh, 1038sh, 1014m, 992s, 960m, 934m, 846w, 831m, 765wb, 742sh, 734m, 728w, 721m, 706m, 672m, 648w, 575vw, 568vw, 341vw.

$C_{18}H_8F_4$ *(KBr disc)*: 1635w, 1594w, 1512s, 1463s, 1459s, 1421s, 1349w, 1266m, 1191w, 1168w, 1107w, 1073s, 1055s, 1011s, 1000sh, 857msh, 851s, 770wsh, 755s, 732m, 718s, 645w, 639m, 614vw, 523w, 428w.

$C_{18}H_4F_8$*(Nujol)*: 1638w, 1630w, 1596w, 1517s, 1365m, 1332w, 1270vwb, 1195w, 1136vw, 1092s, 1077sh, 1055w, 1042sh, 1033s, 952m, 885m, 824m, 774s, 744w, 725s, 659m, 639w, 617w, 458vw, 354vw.

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