

2,2'',3,3'',4,4'',5,5''-Octaphenyl-1,1':4',1''-terphenyl and 2',3',5',6'-tetrafluoro-2,2'',3,3'',4,4'',5,5''-octaphenyl-1,1':4',1''-terphenyl

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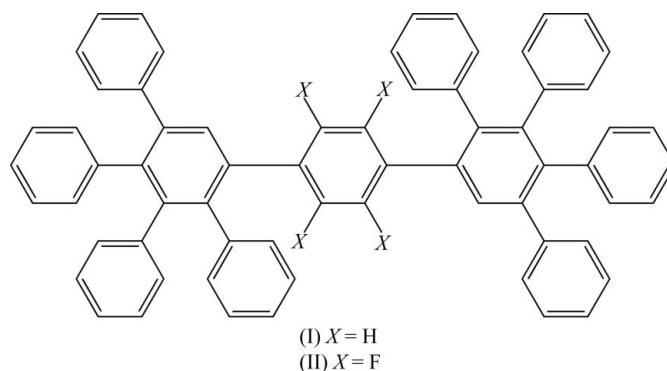
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The title compounds, C₆₆H₄₆, (I), and C₆₆H₄₂F₄, (II), are polyphenylated arylenes synthesized by one-step Diels–Alder cycloaddition reactions. In both structures, all molecules lie on crystallographic inversion centers. In the case of (I), there are two half-molecules present in the asymmetric unit, (IA) and (IB); the geometry of each half-molecule differs principally in the magnitudes of the dihedral angles between mean planes fitted through the central aryl ring and the pendant phenyl rings. The crystal used was a non-merohedral twin, with a refined twin scale factor of 0.460 (8). The dihedral angle between the plane of the central tetrafluorinated ring and the adjacent tetraphenylated ring in (II) is 83.87 (4)°, significantly greater than the dihedral angles of 49.89 (12) and 54.38 (10)° found in the two half-molecules in (IA) and (IB), respectively, and attributed to intermolecular C–H···F hydrogen bonding in (II). Intermolecular C–H···π bonding is found in (I). Two interactions have the C–H bond oriented towards the centroid (C_g) of a butadiene fragment of a phenyl ring; both H···C_g distances are approximately 2.68 Å and the interactions connect adjacent molecules into stacks in the *c*-axis direction. The composition of the stacks alternates, *i.e.* (IA)–(IB)–(IA)–(IB) *etc.* A third, weaker, C–H···π interaction and a phenyl–phenyl close contact connect each end of the long molecular axes of (IB) with an adjacent molecule of (IA) into chains which run perpendicular to the (140) and (1̄40) planes. C–H···F interactions in (II) have the most profound influence on the molecular and crystal structure, the main effect of which is the above-mentioned increase in the dihedral angle between the plane of the central tetrafluorinated ring and the adjacent tetraphenylated ring. C–H···F interactions have refined H···F distances of 2.572 (15)

and 2.642 (16) Å, with approximate C–H···F angles of 123 and 157°, respectively. These form a hydrogen-bonded ribbon structure which propagates in the *b*-axis direction.

Comment

Polyphenylated aromatic molecules are of interest as building blocks in high-performance polymers, such as polyimides, poly(aryl ether)s, poly(ether ketone)s and polysulfones, which possess high glass transition (*T_g*) temperatures, high thermal stability and good mechanical properties and, therefore, have been identified for a variety of applications in government, industry and academia (Yates & Hayes, 2004; Chae & Kumar, 2006). The steric bulk of the phenyl substituents forces the polyphenylene backbone out of conjugation, making these materials insulating and soluble in organic solvents (Berresheim *et al.*, 1999). The addition of fluorine onto the aromatic molecules increases their oxidative stability (Drobny, 2001). The Diels–Alder cycloaddition of bicyclopentadienones with acetylenes has been widely used to produce polyphenylated aromatics and polyarylenes (Stille *et al.*, 1966; Rusanov *et al.*, 2006), including those used as polymer electrolytes for fuel cells (Fujimoto *et al.*, 2005). The former have often been used as model compounds to help understand the regiochemistry in the polymeric forms (Gagnon, Halperin *et al.*, 2010; Gagnon, Maris *et al.*, 2010).



As part of our research we reinvestigated 2,2'',3,3'',-4,4'',5,5''-octaphenyl-1,1':4',1''-terphenyl, (I), which was previously reported (Ried & Bönnighausen, 1960) but without a crystal structure, while 2',3',5',6'-tetrafluoro-2,2'',3,3'',4,4'',-5,5''-octaphenyl-1,1':4',1''-terphenyl, (II), has not been previously reported. The molecular structures of the Diels–Alder adducts (I) and (II) are presented here.

The asymmetric unit of (I) contains two half-molecules; consequently, there are two crystallographically unique molecules, (IA) and (IB), that are generated from these half-molecules by inversion symmetry. Both of these are shown in Fig. 1. An overlay of (IA) with (IB), showing the relative orientations of the various pendant and central phenyl rings, is shown in Fig. 2. Selected dihedral angles for (IA) and (IB), and for related compounds in the Cambridge Structural Database (CSD; Version 5.21, plus four updates; Allen, 2002), are provided in Table 1. Comparison of the dihedral angles for

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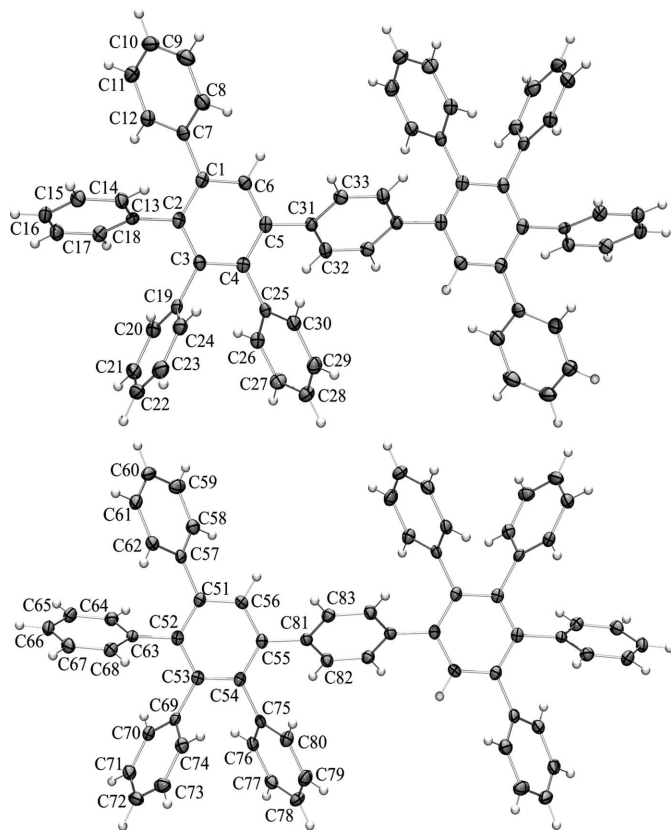


Figure 1

The molecular structures of (IA) (top) and (IB) (bottom), with displacement ellipsoids at the 50% probability level. Unlabeled atoms are related to labeled atoms by crystallographic inversion symmetry.

(I) with PUNVOK in Table 1 reveals that those for (IA) and (IB) are relatively typical except for the 1/4 and 1/5 dihedral angles for (IA), both of which are noticeably elevated. The large difference between 1 and 4 is likely the result of the role of ring 4 in (IA) as an acceptor in a C–H··· π interaction, whereas ring 4 in (IB) is not involved in a similar interaction. Similarly, in (IA), ring 5 is involved in C–H··· π interactions with (IB). In the case of ring 6 in (II), this is involved in hydrogen bonding (discussed later). The values for PUNVEA are included in Table 1 for the purpose of contrast, since in this case C6 of the terphenyl core is substituted (*i.e.* C6-phenyl). All of the dihedral angles in PUNVEA are elevated relative to the rest of Table 1 owing to the presence of additional intramolecular steric crowding caused by the extra phenyl ring. It should also be noted that several of the literature structures are solvates, contain structural disorder, or are charged species with bulky counter-ions; the presence of such additional species in the asymmetric unit can make rationalization of molecular conformation on the basis of intermolecular interactions between chemically equivalent molecules rather difficult. However, in this case, the analysis in Table 1 shows a reasonable degree of consistency between the dihedral angles.

Three unique intermolecular C–H··· π interactions are observed in (I) (Table 2). Two of these, C27–H27···CgA and C79–H79···CgBⁱⁱⁱ [symmetry code: (ii) $x, y, z + 1$], are best described as having the C–H bond oriented towards the

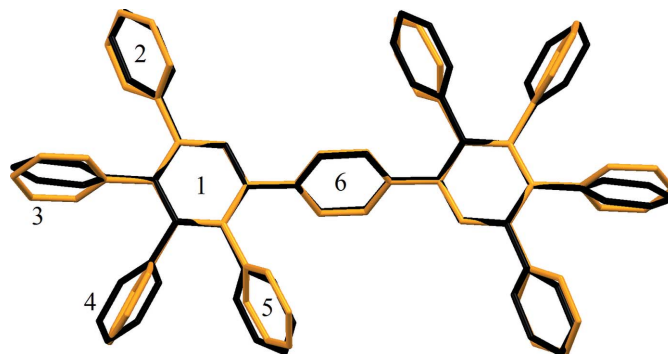


Figure 2

An overlay of molecule A (gray; orange in the electronic version of the paper) with molecule B (black) in (I), formed by a least-squares fit of the six C atoms of ring 1, with an r.m.s. deviation of 0.0377 Å. The ring-numbering system is used to identify angles between least-squares planes.

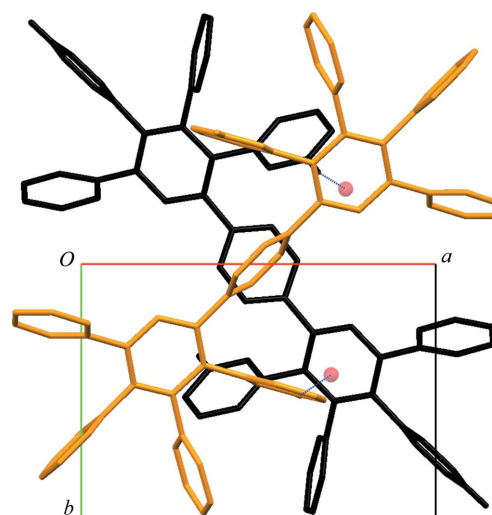
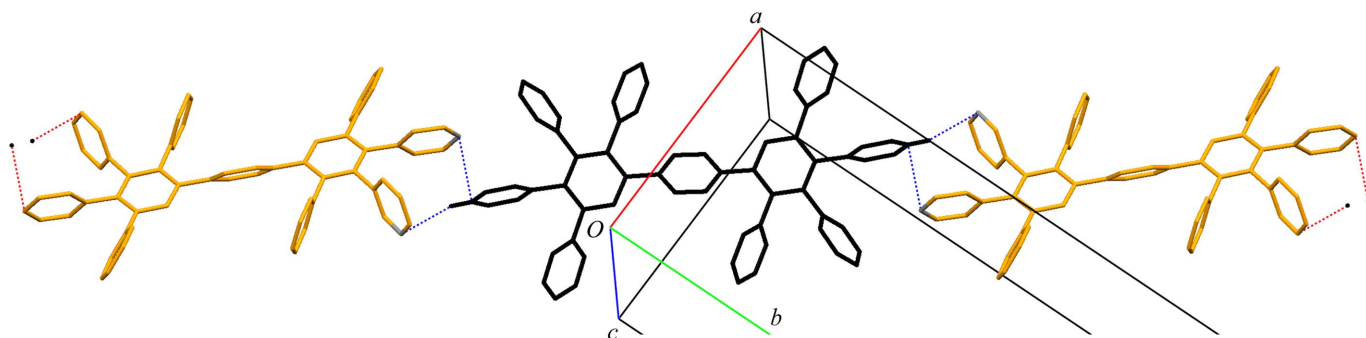


Figure 3

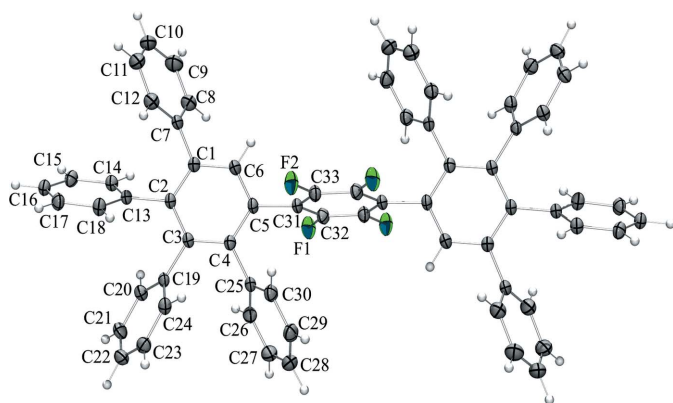
C–H··· π interactions, shown as dashed lines, in (I). The color scheme is the same as used in Fig. 2. The long b axis has been truncated.

centroid of a butadiene fragment of an adjacent phenyl ring. The H27···CgA and H79···CgBⁱⁱⁱ distances are both approximately 2.68 Å, where CgA and CgB are centroids defined in Table 2. These interactions connect adjacent molecules into stacks in the c -axis direction; the composition of the stacks alternates, *i.e.* (IA)–(IB)–(IA)–(IB) *etc.*, and in alternate layers the long molecular axis is rotated by approximately 71° (Fig. 3).

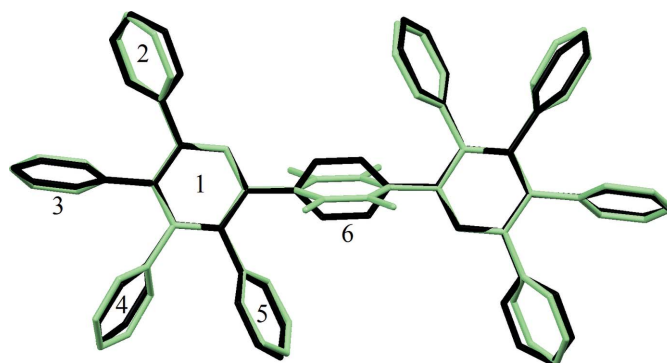
The third interaction is between C66–H66 and the π -electron density above C23ⁱ [symmetry code: (i) $x + 1, -y + \frac{1}{2}, z + \frac{1}{2}$] and is much weaker than the previous two interactions, with an H66···C23ⁱ distance of 2.85 Å. In addition, there is an intermolecular close contact between adjacent phenyl rings at the ends of the molecules. The C66···C16ⁱ distance is 3.255 (3) Å, shorter than the van der Waals sum of 3.40 Å for two C atoms. The marked increase in the 1/4 dihedral angle in (IA) is attributed to these two interactions. These two interactions also connect each end of the long molecular axes of (IB) with an adjacent molecule of (IA) into (IA)–(IB)–(IA)–

**Figure 4**

The C—H... π -bonded chain in (I). C—H... π interactions are shown as dashed lines. The color scheme is the same as used in Fig. 2. The long b axis has been truncated.

**Figure 5**

Twice the asymmetric unit of (II), with displacement ellipsoids at the 70% probability level. Unlabeled atoms are related to labeled atoms by crystallographic inversion symmetry.

**Figure 6**

An overlay of (IB) and (II), fitted in the same way as for Fig. 2, with an r.m.s. deviation of 0.0278 Å. (II) is shown in gray (green in the electronic version of the paper).

(IB) *etc.* chains oriented perpendicular to the (140) and $(\bar{1}40)$ planes (an example of one such chain is given in Fig. 4). The complete description of the crystal structure of (I) is a combination of one-dimensional stacks and one-dimensional chains, which combine to give two sets of interpenetrated three-dimensional networks of intermolecularly associated molecules.

The asymmetric unit of (II) contains just one half-molecule of the tetrafluorinated adduct. A whole molecule of (II), consisting of two asymmetric units related by crystallographic inversion symmetry, is shown in Fig. 5 and selected dihedral angles are given in Table 1. The $1/6$ dihedral angle in (II) is $83.87(4)^\circ$, while the corresponding angles in (IA), (IB) and perfluorobiphenyl (Nae, 1979) are $49.89(12)$, $54.38(3)$ and 59.6° , respectively. As shown in Fig. 6, larger $1/6$ values correspond to a more twisted and less planar terphenyl core. A broader survey of related compounds (Table 1) shows that the $1/6$ angle for (II) is unusual, over 20° greater than all others. This can be attributed to the participation of the F atoms in C—H...F hydrogen bonding, as discussed below. The aromatic C—F bond distances are consistent with typical values for *ortho*-F atoms, approximately 0.02 Å shorter than other types of Ar—F bond (Ar is aryl; Allen *et al.*, 1987). There is no crystal structure with which to compare the

tetrafluorinated terphenyl model system, and the tetrafluorinated pentaphenylated version has not been synthesized or reported in the literature.

The presence of C—H...F interactions in (II) is a notable feature (Fig. 7 and Table 3). All F atoms are involved in these interactions, which form a hydrogen-bonded ribbon structure which propagates in the b -axis direction. The shortest F...F distance (3.19 Å) is longer than the van der Waals sum for two F atoms (2.91 Å) and so F...F close contacts are unlikely in this structure. Therefore, the formation of C—H...F interactions in (II) is the main driving force behind the significant nonplanar topology of the terphenyl core, as evidenced by the magnitude of the $1/6$ angle in (II). By contrast, C—H... π interactions are hindered. The only exception to this is a long H27...C16ⁱⁱⁱ interaction (details and symmetry code are in Table 3) which is between adjacent ribbons in the crystal packing and has no role in the topology of the terphenyl core.

It is well known that *meta*- and *para*-substituted biphenyls and biphenyl itself have a planar configuration (Bastiansen, 1949), whereas extensive studies on terphenyl show it to be nonplanar at low temperature (Baudour *et al.*, 1977, 1986). For polyphenylated terphenyl and similar compounds, Gagnon, Maris *et al.* (2010) noted that such compounds generally lack aromatic interactions, attributed to the nonplanar topology of the molecules, which hinders close packing. Here we show that

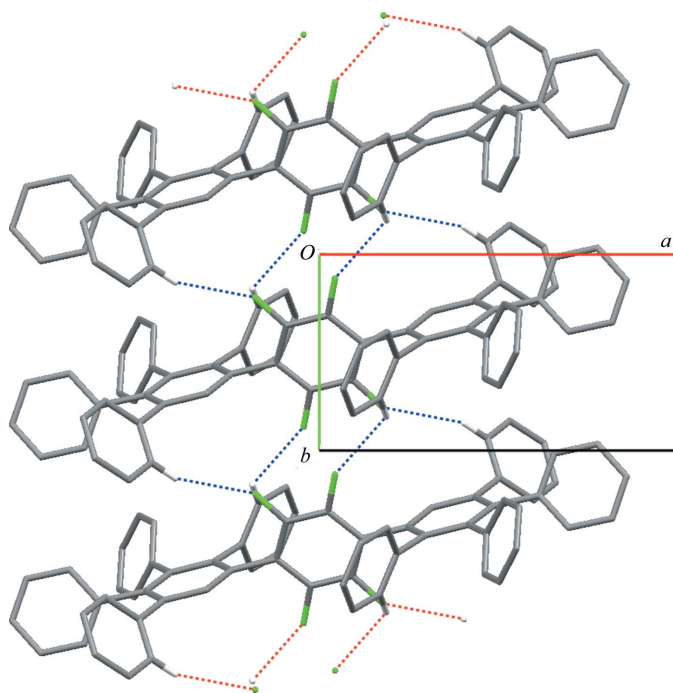


Figure 7
C—H...F and π – π interactions (dotted lines) in (II). The *a* axis has been truncated.

close packing interactions do indeed exist between aromatic rings in this type of compound, although they are few in number when compared with the number of aromatic rings present. Indeed, quite dramatic changes in both molecular conformation and three-dimensional structure can be effected by fluorine substitution at the central aryl ring, which result in newly formed C—H...F intermolecular interactions, an entirely different molecular conformation and hence a quite different crystal structure.

Experimental

1,4-Diethynylbenzene was obtained from Aldrich and purified by sublimation before use. 2,3,4,5-Tetraphenylcyclopentadienone (tetracyclone) was synthesized according to published methods (Johnson & Grummitt, 1943) and crystallized from a mixture of ethanol and benzene. 1,4-Diethynyl-2,3,5,6-tetrafluorobenzene was synthesized according to previous methods and sublimed before use (Neenan & Whitesides, 1988). Diels–Alder adducts (I) and (II) were obtained by the reaction between 1,4-diethynylbenzene (57 mg, 0.455 mmol) or 1,4-diethynyl-2,3,5,6-tetrafluorobenzene (90 mg, 0.455 mmol), respectively, with tetracyclone (350 mg, 0.910 mmol) under argon in diphenyl ether (5 ml) in a round-bottomed flask at 423 K for 24 h or until the color changed from dark purple to yellow or pink, respectively. The products were cooled to room temperature, precipitated with acetone (100 ml) and vacuum filtered; the solid was washed with acetone (10 ml) and dried in a vacuum oven at 333 K for 24 h, affording white solids in yields of 78 and 84%, respectively. In both cases, crystals suitable for X-ray analysis were grown from solutions (10–20 mg) in deuterated dimethyl sulfoxide (DMSO-*d*₆, 5–10 ml). Details of nuclear magnetic resonance assignments and high-resolution mass spectrometry are given in the archived CIF.

Table 1
Selected dihedral angles (°) in (I), (II) and related compounds.

Dihedral angles are between least-squares planes fitted through all non-H atoms of the pendant phenyl rings and the central aryl ring; rings are numbered according to the system described in Fig. 2. Compounds with multiple entries contain more than one pentaphenylated component. Dihedral angles for (IA), (IB) and (II) were calculated using *SHELXTL* (Sheldrick, 2008*b*); all other dihedral angles were determined using *PLATON* (Spek, 2009). Ring 6 of PUNVEA contains an extra phenyl ring, 7; the dihedral angle 6/7 in PUNVEA is 85.59 (6)°.

Compound	1/2	1/3	1/4	1/5	1/6
(IA)	58.66 (8)	67.55 (7)	86.08 (6)	73.56 (7)	49.89 (12)
(IB)	49.24 (8)	65.82 (7)	64.44 (6)	67.21 (6)	54.38 (10)
(II)	64.10 (3)	60.42 (4)	55.83 (4)	66.17 (3)	83.87 (4)
BIJBEC ^a	61.53 (17)	56.17 (15)	67.30 (16)	67.56 (16)	51.33 (15)
	45.63 (17)	62.99 (15)	65.35 (16)	64.22 (17)	53.07 (15)
EGIKUA ^{†b}	55.0 (3)	55.9 (3)	64.3 (3)	54.4 (3)	46.7 (3)
	55.6 (3)	66.6 (3)	63.3 (3)	60.1 (3)	48.3 (3)
	56.5 (4)	61.1 (3)	66.4 (3)	61.1 (3)	44.9 (3)
	60.7 (4)	61.4 (3)	64.1 (4)	57.2 (4)	38.7 (3)
	62.2 (3)	60.2 (4)	68.0 (4)	54.2 (4)	42.3 (3)
	55.0 (3)	55.9 (3)	64.3 (3)	54.4 (3)	46.7 (3)
IQESI ^{†c}	50.76 (4)	54.20 (4)	71.12 (4)	63.68 (3)	47.59 (3)
	49.17 (3)	58.47 (3)	63.13 (3)	57.10 (4)	50.46 (3)
NILQAB ^d	48.63 (17)	64.88 (15)	66.37 (15)	67.75 (15)	50.16 (14)
PUNVIE ^c	56.09 (7)	58.12 (7)	72.92 (7)	58.13 (7)	57.18 (7)
PUNVOK ^c	49.83 (8)	63.93 (8)	62.78 (9)	61.33 (9)	60.35 (8)
PUNVEA ^c	69.19 (6)	77.36 (6)	80.79 (8)	85.67 (6)	61.26 (5)

[†] Structure contains solvent or counter-ion. References for CSD refcodes: (a) Grebel-Koehler *et al.* (2003); (b) Bauer *et al.* (2002); (c) Türp *et al.* (2011); (d) Chen *et al.* (2007); Gagnon, Maris *et al.* (2010).

Table 2
Intermolecular π -interactions (Å, °) in (I).

CgA is the centroid of atoms C52/C53/C54/C55 and *CgB* is the centroid of atoms C1/C4/C5/C6.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C27—H27... <i>CgA</i>	0.95	2.68	3.58	157
C66—H66...C23 ⁱ	0.95	2.85	3.701 (3)	150
C79—H79... <i>CgB</i> ⁱⁱ	0.95	2.68	3.61	165

Symmetry codes: (i) $x + 1, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $x, y, z + 1$.

Compound (I)

Crystal data

$C_{66}H_{46}$	$V = 4597.1 (14) \text{ \AA}^3$
$M_r = 839.03$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 11.674 (2) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$b = 33.336 (6) \text{ \AA}$	$T = 100 \text{ K}$
$c = 11.816 (2) \text{ \AA}$	$0.35 \times 0.09 \times 0.08 \text{ mm}$
$\beta = 91.479 (3)^\circ$	

Data collection

Bruker Kappa APEXII DUO CCD diffractometer	40040 measured reflections
Absorption correction: multi-scan (<i>TWINABS</i> ; Sheldrick, 2008 <i>a</i>)	11292 independent reflections
$T_{\min} = 0.977, T_{\max} = 0.995$	8284 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.068$
	$\theta_{\max} = 23.6^\circ$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$	596 parameters
$wR(F^2) = 0.107$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\max} = 0.16 \text{ e \AA}^{-3}$
11292 reflections	$\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$

Table 3Intermolecular hydrogen bonding and π -interactions (\AA , $^\circ$) in (II).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C30—H30 \cdots F1 ⁱ	0.981 (16)	2.572 (15)	3.2084 (11)	122.6 (11)
C8—H8 \cdots F2 ⁱⁱ	0.967 (16)	2.642 (16)	3.5517 (12)	157.0 (12)
C27—H27 \cdots C16 ⁱⁱⁱ	1.000 (16)	2.825 (16)	3.6927 (14)	145.5 (12)

Symmetry codes: (i) $x, y + 1, z$; (ii) $x, y - 1, z$; (iii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.**Compound (II)***Crystal data*

$C_{66}H_{42}F_4$	$V = 2329.89$ (19) \AA^3
$M_r = 911.00$	$Z = 2$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 19.7494$ (9) \AA	$\mu = 0.09$ mm^{-1}
$b = 6.1217$ (3) \AA	$T = 100$ K
$c = 20.4762$ (10) \AA	$0.31 \times 0.19 \times 0.16$ mm
$\beta = 109.754$ (2) $^\circ$	

Data collection

Bruker Kappa APEXII DUO CCD diffractometer	55308 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2008a)	6808 independent reflections
$T_{\min} = 0.974$, $T_{\max} = 0.986$	5932 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	400 parameters
$wR(F^2) = 0.112$	All H-atom parameters refined
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.44$ e \AA^{-3}
6808 reflections	$\Delta\rho_{\text{min}} = -0.21$ e \AA^{-3}

For (I), the crystal used was found to exhibit non-merohedral twinning, which was handled by a combination of *CELL_NOW* (Sheldrick, 2004) and *TWINABS* (Sheldrick, 2008a), with successive refinement of the unit-cell parameters by *SAINT* (Bruker, 2007). The twin law is $100/0\bar{1}0/00\bar{1}$ and the refined twin scale factor is 0.460 (8) (3782 unique reflections involve domain 1, 3715 unique reflections involve domain 2 and 4039 overlapped reflections involve both domains). Diffraction was only observed to a resolution of 0.89 \AA and so the data set was truncated at this limit. H atoms were initially located from a difference Fourier map, but were then constrained to ride on their parent atoms, with a C—H distance of 0.95 \AA and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. For (II), all H atoms were located in a difference map and were freely refined. C—H distances lie in the range 0.962 (16)–1.015 (14) \AA . The largest residual peak is 0.69 \AA from atom C33.

For both compounds, data collection: *APEX2* (Bruker, 2007). Cell refinement: *CELL_NOW* (Sheldrick, 2004) and *SAINT* (Bruker, 2007) for (I); *SAINT* for (II). For both compounds, data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008b); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *Mercury*

(Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL*, *publCIF* (Westrip, 2010) and local programs.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: EG3079). Services for accessing these data are described at the back of the journal.

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