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1,6,11,18,24,27,52,55-Octakis(trifluoromethyl)-1,6,11,18,24,27,52,55-octahydro-(C_{60}-I_h)[5,6]fullerene

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.123
Data-to-parameter ratio = 12.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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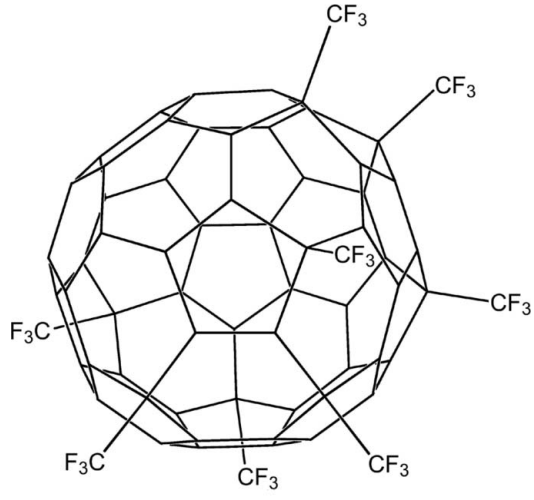
The title compound, $C_{68}F_{24}$, is one of five isomers of $C_{60}(CF_3)_8$. It has an idealized I_h C_{60} core with the eight CF_3 groups arranged on an asymmetric *para–para–para–meta–para* (p^3mp) ribbon of six edge-sharing $C_6(CF_3)_2$ hexagons plus an isolated *p*-$C_6(CF_3)_2$ hexagon. There are no cage Csp^3–Csp^3 bonds. There are intramolecular F···F contacts between pairs of neighboring CF_3 groups that range from 2.582 (3) to 2.647 (3) Å.

Comment

The high-temperature reaction of C_{60} with CF_3I or $AgCF_3COO$ followed by sublimation at 673–773 K and HPLC purification has yielded one isomer each of $C_{60}(CF_3)_2$ and $C_{60}(CF_3)_4$ (Goryunkov *et al.*, 2003), two isomers of $C_{60}(CF_3)_6$ (Goryunkov *et al.*, 2003; Kareev, Shustova *et al.*, 2006), at least four isomers of $C_{60}(CF_3)_{10}$ (Kareev *et al.*, 2005; Kareev, Lebedkin, Miller *et al.*, 2006; Kareev, Lebedkin, Popov *et al.*, 2006), and one isomer of $C_{60}(CF_3)_{12}$ (Troyanov *et al.*, 2006). In a similar fashion, we have now isolated five isomers of $C_{60}(CF_3)_8$. The title compound, (I), is one of these five new compounds and we report its crystal structure here.



(I)

The structure of (I) (Fig. 1) comprises an idealized I_h C_{60} cage with eight Csp^3 atoms at positions 1, 6, 11, 18, 24, 27, 53 and 56 (IUPAC nomenclature), each of which is attached to a CF_3 group. Each Csp^3 cage atom is adjacent to three Csp^2 cage atoms. The CF_3 groups are arranged on one isolated *para*-$C_6(CF_3)_2$ hexagon and a *para–para–para–meta–para* ribbon (a p^3mp ribbon) of edge-sharing $C_6(CF_3)_2$ hexagons (see Schlegel diagram in Fig. 1). The shared edges in the ribbon of

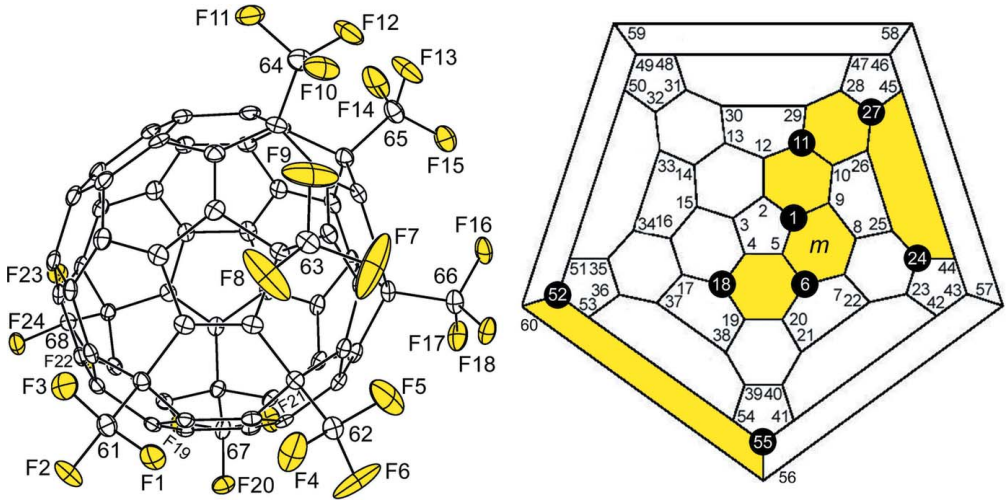


Figure 1
Left: The molecular structure of (I), showing the atom-labeling scheme and with displacement ellipsoids drawn at the 50% probability level. Right: Schlegel diagram of (I), showing the C_{60} core C-atom numbers (each core C atom bearing a CF_3 group is depicted as a black circle) and the p^3mp,p addition pattern [the *meta*-$C_6(CF_3)_2$ hexagon is indicated by the letter *m*].

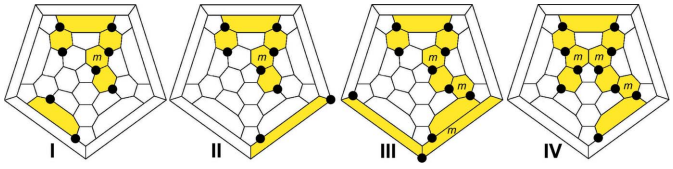


Figure 2
Schlegel diagrams of (I), (II), (III) and (IV), showing the location of the R_f groups as black circles on the ribbons of *meta*- and *para*-$C_6(R_f)_2$ edge-sharing hexagons [*meta*-$C_6(R_f)_2$ hexagons are indicated by the letter *m*]. All four compounds have a p^3mp ribbon as part of their structures [compound (IV) has two such ribbons in its structure].

hexagons are $Csp^3—Csp^2$ bonds. Thus, any pair of adjacent hexagons along the ribbon have a common CF_3 group. There are F···F intramolecular contacts between pairs of neighboring CF_3 groups that range from 2.582 (3) to 2.647 (3) Å.

Fig. 2 shows the Schlegel diagrams for (I) and for the related addition patterns of C_1-p^3mp,p-$C_{60}(C_2F_5)_8$, (II) (Kareev, Kuvychko *et al.*, 2006), C_1-$p^3mpmpmp$-$C_{60}(CF_3)_{10}$, (III) (Kareev, Lebedkin, Miller *et al.*, 2006), and C_1-pmp^3mpmp-$C_{60}(CF_3)_{10}$, (IV) (Kareev *et al.*, 2005). The structures of all four compounds include the p^3mp ribbon or ribbon fragment of six edge-sharing $C_6(R_f)_2$ hexagons that is also believed to be the addition pattern for C_1-$C_{60}(CF_3)_6$ (Goryunkov *et al.*, 2003). The four shortest cage C—C bonds (Å) are C4—C5 [1.345 (3)], C7—C8 [1.351 (4)], C9—C10 [1.358 (4)] and C56—C60 [1.357 (4)]. Significantly, the C4–C5 and C9—C10 bonds are pentagon–hexagon junctions, which are the longer of the two types of C—C bonds in C_{60}.

Experimental

The synthesis of (I) was accomplished by heating C_{60} in a stream of CF_3I at 733 K as previously reported (Kareev *et al.*, 2005). Crystals of the HPLC-purified compound were grown by slow evaporation of a saturated toluene solution.

Crystal data

$C_{68}F_{24}$
M_r = 1272.68
Monoclinic, $P2_1/c$
a = 17.4108 (13) Å
b = 9.7708 (8) Å
c = 24.5142 (18) Å
β = 92.589 (4)°
V = 4166.0 (6) Å^3
Z = 4
D_x = 2.029 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 100 (1) K
Plate, red
0.30 × 0.12 × 0.03 mm

Data collection

Bruker Kappa-APEX-II diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.945, T_{max} = 0.995
64785 measured reflections
10338 independent reflections
6471 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.123
S = 1.01
10338 reflections
830 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 4.7196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.00067 (18)

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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