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Elusive Fullerenes

Discovering and Verifying Elusive Fullerene Cage Isomers: Structures of C_2-p^{11}-(C_{74}-D_{3h})(CF_3)$_{12}$ and C_2-p^{11}-(C_{78}-D_{3h}(5))(CF_3)$_{12}$**

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We recently reported the trifluoromethylation of a mixture of sublimable but insoluble and largely unknown fullerenes at 500 °C and the isolation and NMR characterization of single isomers of stable, soluble $C_{2n}(CF_3)_{12}$ derivatives for $C_{2n} = C_{74}$-D_{3h}, C_{76}-T_d(2), C_{78}-D_{3h}(5), C_{80}-C_{2v}(5), and C_{82}-C_2(3).[1] In each case, a combination of 1D and 2D NMR data and DFT calculations narrowed down the billions of possible isomers to one probable isomer. Among the predicted structures were C_2-p^{11}-(C_{74}-D_{3h})(CF_3)$_{12}$ and C_2-p^{11}-(C_{78}-D_{3h}(5))(CF_3)$_{12}$, both with a continuous ribbon of 11 edge-sharing *para*-$C_6(CF_3)_2$ hexagons.[1] We now confirm the proposed structures for both compounds by single-crystal X-ray diffraction. These are the first X-ray structures of any hollow or endohedral derivative of the C_{74}-D_{3h} or C_{78}-D_{3h}(5) cages that 1) do not exhibit "general disorder of all … cage [C atoms]" (see the description of the crystal structure of La@$C_{74}(C_6H_3Cl_2)$[2]) or 2) were not refined using a rigid-body DFT-optimized carbon cage (see the refinement of the structures of Ba@(C_{74}-D_{3h})[3] and Sc_3N@(C_{78}-D_{3h}(5))[4]). In addition, the precision of the C_{74} structure permits a meaningful analysis of the C−C distances, C−CF_3 distances, and F-C-C-C torsion angles.

Several high-quality X-ray structures of fullerene derivatives with two or more CF_3 or C_2F_5 groups have been reported since the first one was published in mid-2005;[5] most exhibit unprecedented C_1-symmetry addition patterns.[6] High-temperature perfluoroalkylation results in the isolation of a small number of the many possible isomers that probably form during the radical addition of multiple CF_3 and C_2F_5 groups.[5,7,8] In addition, it has been shown that trifluoromethylation of a mixture containing Y@C_{82} and Y_2@C_{80} facilitated the isolation of purified Y_2@C_{80} and, at the same time, converted paramagnetic Y@C_{82} into two stable, soluble, and diamagnetic isomers of Y@$C_{82}(CF_3)_5$.[9] The two X-ray structures reported here demonstrate that high-temperature trifluoromethylation can also lead to the discovery or verification of elusive fullerene cages.

Hollow C_{74} was observed in sublimable fractions of soots in 1993,[10] and its electron affinity (3.28(7) eV) was measured in 1996.[11] There is only one possible isomer for C_{74} that follows the isolated-pentagon rule, C_{74}-D_{3h}.[12] The exohedral derivative $C_{74}F_2$ was observed by Knudsen-cell mass spectrometry in 1997.[13] Diener and Alford reported the purification of C_{74} in 1998,[14] and in 2004 we reported the preparation and ^{19}F NMR spectrum of its first isolable derivative, $C_{74}F_{38}$, the ^{19}F NMR spectrum of which was consistent with D_3 symmetry. This provided the first experimental evidence that hollow C_{74} probably has D_{3h} symmetry.[15] Endohedral C_{74} compounds have been investigated, and their ^{13}C NMR spectra suggest that the M@C_{74} cage has the D_{3h} structure as well (see references [2] and [3] and references therein).

The X-ray crystal structure of $C_{74}(CF_3)_{12}$ is shown in Figure 1 a.[16] The complete thermal ellipsoid plot, numbering, and packing diagrams are available.[6] The $C_{74}(CF_3)_{12}$ molecule has crystallographic C_2 symmetry. The C_2 axis is the only remaining symmetry element of the original D_{3h} cage after the 12 CF_3 groups have been added to give a ribbon of 11 C(sp^3)−C(sp^2) edge-sharing *p*-$C_6(CF_2)_2$ hexagons (see Schlegel diagram in Figure 1 c). The estimated standard deviations for individual cage C−C distances range from 0.0017 to 0.0020 Å (Table 1). A network of F···F contacts between hexagon-sharing CF_3 groups range in distance from 2.6322(17) to 2.8785(14) Å and give rise to the time-averaged, through-space Fermi-contact $^7J_{FF}$ values of 12–15 Hz that are evident in the ^{19}F NMR spectrum of this compound.[1,6]

The (*para*)^{11} (i.e., p^{11}) ribbon of *p*-$C_6(CF_3)_2$ hexagons in C_2-$C_{74}(CF_3)_{12}$ is the longest *para*-only ribbon in fullerene-$(CF_3)_n$ compounds reported to date (Figure 1 d). There are six X-ray structures of fullerenes with exactly 12 CF_3 groups.[6] One has a closed loop of alternating *p*- and *m*-$C_6(CF_3)_2$ hexagons, S_6-$(pm)^6$(loop)-$C_{60}(CF_3)_{12}$.[7] The others have single-ribbon addition patterns: C_2-p^{11}-$C_{74}(CF_3)_{12}$, C_2-p^{11}-C_{78}-$(CF_3)_{12}$ (see below), two isomers of C_1-p^7mp,p-$C_{70}(CF_3)_{12}$,[17,18]

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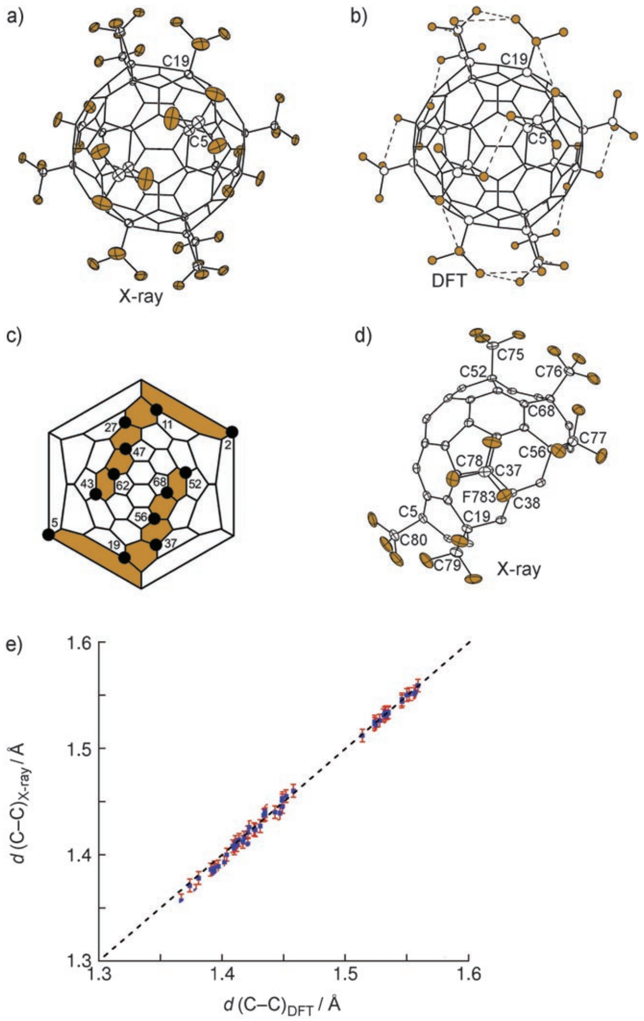

Figure 1. C_2-p^{11}-(C_{74}-D_{3h})$(CF_3)_{12}$: a) the X-ray structure (50% probability ellipsoids for selected atoms; cocrystallized *p*-xylene molecules omitted for clarity; F brown), b) the DFT-optimized structure, and c) a Schlegel diagram. The F···F contacts between CF_3 groups are marked in the DFT structure in (b) (DFT distances: 2.676–2.864 Å; X-ray distances: 2.632(2)–2.878(2) Å). d) The F783-C78-C37-C38 torsion angle visible in the fragment of the structure showing half of the p^{11} ribbon is 0.9(1)°. e) The plot shows the correlation between the cage C–C distances in the X-ray structure vs. those in the DFT structure (±3σ error bars).

Table 1: Interatomic distances [Å] and angles [°] for C_2-p^{11}-(C_{74}-D_{3h})-$(CF_3)_{12}$ from the X-ray structure and DFT-optimized structure.

Parameter	X-ray	DFT	CF_3 locants
	F···F distance		
F751···F762	2.6325(15)	2.690	43···62
F761···F772	2.7011(16)	2.741	62···47
F771···F782	2.8785(14)	2.864	47···27
F771···F783	2.7595(15)	2.766	47···27
F781···F792	2.6823(14)	2.745	27···11
F782···F792	2.8079(14)	2.853	27···11
F791···F802	2.6223(17)	2.676	11···2
F801···F811	2.639(2)	2.685	2···5
	$F_3C···CF_3$ distance		
C75···C76	3.962(2)	4.013	43···62
C76···C77	4.213(2)	4.309	62···47
C77···C78	4.024(2)	4.053	47···27
C78···C79	3.986(2)	4.042	27···11
C79···C80	4.313(2)	4.340	11···2
C80···C81	3.875(2)	3.938	2···5
	F-C-C-C torsion angle		
F751-C75-C43-C60	52.0(1)	52.4	43
F762-C76-C62-C45	24.7(1)	23.4	62
F772-C77-C47-C46	47.1(1)	54.0	47
F782-C78-C27-C28	0.8(1)	0.9	27
F791-C79-C11-C10	56.4(1)	51.5	11
F802-C80-C2-C1	33.6(1)	33.0	2

and C_1-$p^3(mp)^4$-$C_{60}(CF_3)_{12}$.[19] (Note that all but the latter compound have one CF_3 group on each of the 12 pentagons.)

We previously reported DFT-optimized structures of C_2-p^{11}-(C_{74}-D_{3h})$(CF_3)_{12}$ and C_2-p^{11}-(C_{78}-$D_{3h}(5)$)$(CF_3)_{12}$ obtained using the PBE functional[20] and the PRIRODA[21] quantum-chemical package.[1] A plot shows the agreement between the cage C–C distances of the X-ray and DFT structures of C_2-p^{11}-$C_{74}(CF_3)_{12}$ (Figure 1e). (We also optimized the structure at the B3LYP/6-31G* level and found virtually no differences.[6]) Table 1 lists F···F distances, $F_3C···CF_3$ distances between hexagon-sharing CF_3 groups, and F-C-C-C torsion angles from the X-ray and the DFT structures. The latter parameter indicates the conformation of each CF_3 group with respect to the fullerene cage. (The torsion angles in Table 1 are the smallest of the torsion angles that a CF bond makes with the relevant underlying cage hex-hex junction; an angle of 60° is defined as staggered, and an angle of 0° is defined as eclipsed.) The agreement for these structural parameters is also very good, including a significant prediction that the CF_3 group at C78 or C78′, the fourth CF_3 group from either end of the ribbon, is eclipsed with a torsion angle of only 1° (see Figure 1d). The significance is that F-C-C-C torsion angles in fullerene$(CF_3)_n$ structures appear to be correlated with $-\delta$ values in the ^{19}F NMR spectrum: it was proposed that a $-\delta$ value <60 indicates that the CF_3 group has an eclipsed or nearly eclipsed conformation,[5,9] and the multiplet for the C27 and C37 CF_3 groups has a $-\delta$ value of 55.2.[6] This correlation and other CF_3-addition-pattern principles were used to predict the probable structures of the $C_{2n}(CF_3)_{12}$ compounds from the many possible isomers ($2n = 74, 76, 78, 80$, and 82).[1] Therefore, the X-ray structures of C_2-$C_{74}(CF_3)_{12}$ and C_2-$C_{78}(CF_3)_{12}$ reported here are the first unambiguous verifications that the correlation between torsion angle and NMR chemical shift, and other addition-pattern principles are probably valid in general. This gives added confidence that the predictions of the cage isomer and addition pattern for C_s-p^9(loop),p^2-(C_{76}-$T_d(2)$)$(CF_3)_{12}$, C_s-p^{10}(loop),p-(C_{80}-$C_{2v}(5)$)$(CF_3)_{12}$, C_2-p^{11}-(C_{82}-$C_2(5)$)$(CF_3)_{12}$, and C_2-p^5,p^5-(C_{82}-$C_2(3)$)$(CF_3)_{12}$ are correct.[1]

Each -*pmp*- ribbon sequence in the $C_{60,70}(CF_3)_n$ structures results in at least one very short pent-hex junction, and these are frequently the shortest cage C–C bonds in the compound. For example, there are three pent-hex junctions in C_1-$p^3mpmpmp$-$C_{60}(CF_3)_{10}$ that range from 1.354(1) to 1.358(1) Å,[22] and the two shortest pent-hex junctions in C_1-

$p^3mpmpmpmp$-$C_{60}(CF_3)_{12}$ are 1.347(2) and 1.350(2) Å.[19] In contrast, the shortest pent-hex junction in all-*para* C_2-$C_{74}(CF_3)_{12}$ is 1.400(2) Å, more than 20σ longer than 1.358 Å.

Neither C_{78}-$D_{3h}(5)$[12] nor any exohedral derivative thereof had been observed experimentally before the synthesis of this compound.[1] Its X-ray structure, Schlegel diagram, and two DFT structures are shown in Figure 2.[23] A thermal ellipsoid plot and complete numbering are available.[6] The structure of this compound proved to be difficult to refine satisfactorily. Two data sets were obtained for crystals from different crystallization batches, and both data sets yielded the same overall structure for the fullerene molecule. However, standard refinement in each case led to physically unreasonable atomic displacement parameters for some atoms in the fullerene cage and chemically unreasonable electron density in parts of the solvent-occupied regions of the structure. The best residual indices were obtained from a model for which the program SQUEEZE[24] was used to fill the disordered solvent regions; we report this model for C_2-p^{11}-$(C_{78}$-$D_{3h}(5))$-$(CF_3)_{12}$ here.

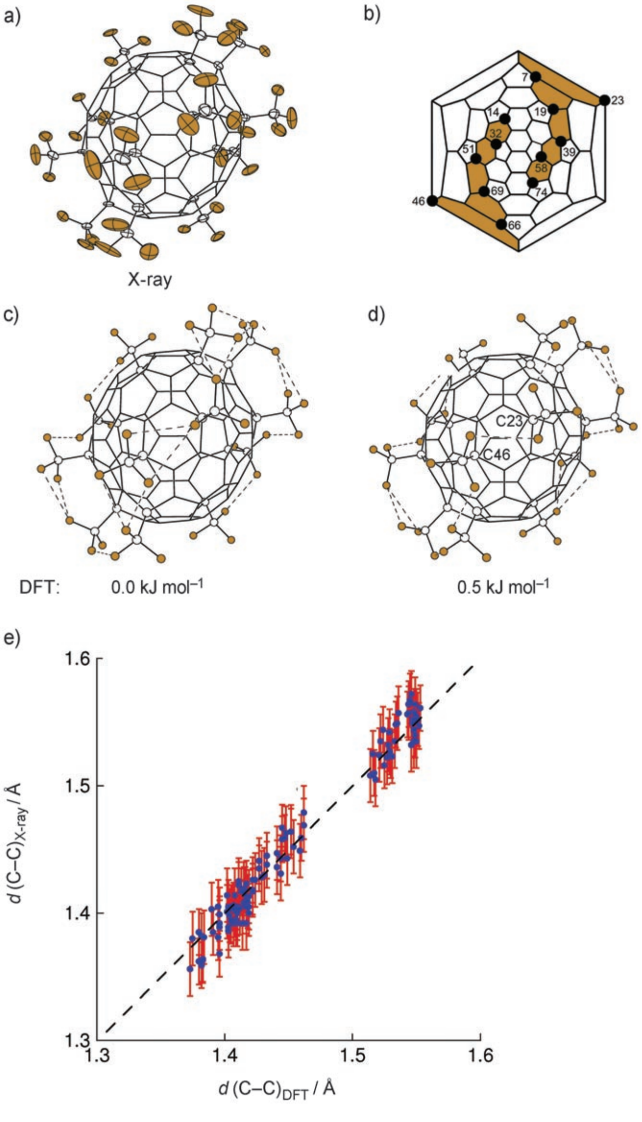

Figure 2. C_2-p^{11}-$(C_{78}$-$D_{3h}(5))(CF_3)_{12}$: a) X-ray structure (50% probability ellipsoids; F brown), b) a Schlegel diagram, and c,d) DFT-optimized structures which differ only in the relative conformations of the CF_3 groups and have an energy difference of only 0.5 kJ mol^{-1}. The intramolecular F···F contacts between hexagon-sharing CF_3 groups are shown in the DFT structures and range from 2.576 to 2.902 Å in the 0.0 kJ mol^{-1} isomer and from 2.649 to 2.898 Å in the 0.5 kJ mol^{-1} isomer (the corresponding distances in the X-ray structure range from 2.479(6) to 2.987(6) Å). e) The plot shows the correlation between the cage C–C distance in the X-ray structure vs. that the DFT structure (at 0.0 kJ mol^{-1}; ±3σ error bars).

Despite the refinement problems for this structure, there is no doubt that it consists of a C_{78}-$D_{3h}(5)$ cage with 12 CF_3 groups forming a ribbon of 11 p-$C_6(CF_3)_2$ hexagons (Figure 2). Although the estimated standard deviations for individual cage C–C bond lengths are larger than for C_2-$C_{74}(CF_3)_{12}$, the plot of the X-ray vs. DFT C–C distances in Figure 2e shows a good correlation between experiment and theory.

The p^{11} ribbon is not even approximately C_2 symmetric with respect to the CF_3 conformations. For example, in the X-ray structure the CF_3 groups attached to C23 and C46 have F-C-C-C torsion angles of 15.5° and 51.8° (av 33.7°), and the CF_3 groups attached to C32 and C58 have torsion angles of 28.0° and 17.3° (av 22.7°). The latter pair of CF_3 groups has the smallest average torsion angle for any pair in this compound. Accordingly, none of the ^{19}F NMR multiplets for C_2-C_{78}-$(CF_3)_{12}$ have $-\delta$ values below 60.[1,6] For comparison, in the structures calculated by DFT (minimum-energy conformational isomer and that 0.5 kJ mol^{-1} higher in energy (Figures 2c and 2d)) the smallest average torsion angle, averaged over both conformational isomers, is 24.8° for the pair of CF_3 groups attached to C32 and C58. The agreement is very good.

Trifluoromethylation has emerged as a powerful tool for the conclusive identification of previously "unknown" fullerene cages, as exemplified here by the structural characterization of C_2-$(C_{74}$-$D_{3h})(CF_3)_{12}$ and C_2-$(C_{78}$-$D_{3h}(5))(CF_3)_{12}$. Attempts to obtain higher-quality crystals of the latter compound and of the other hollow higher fullerenes with 12 CF_3 groups are continuing in our laboratories.

Experimental Section

The two compounds were prepared as previously described from a mixture of sublimed, insoluble higher fullerenes and CF_3I at 500 °C[1] and were crystallized from p-xylene. X-ray diffraction data were obtained for C_2-$C_{74}(CF_3)_{12}$ in Fort Collins using a Bruker Kappa APEX II CCD diffractometer ($Mo_{K\alpha}$ radiation ($\lambda = 0.71073$ Å), graphite monochromator)). X-ray diffraction data were obtained for C_2-$C_{78}(CF_3)_{12}$ in Berlin using a Bruker XPS CCD diffractometer ($Mo_{K\alpha}$ radiation ($\lambda = 0.71073$ Å), graphite monochromator)). In both cases, the semiempirical absorption correction was applied using SADABS.[25] The structures were refined using SHELTXL[26] and, for C_2-$C_{78}(CF_3)_{12}$, the program SQUEEZE was employed.[24] DFT calculations were performed as previously described.[1,8,9]

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[16] Crystals of C_2-p^{11}-(C_{74}-D_{3h})(CF_3)$_{12}$·3(p-xylene) were grown in Fort Collins by slow evaporation of a saturated p-xylene solution: $0.19 \times 0.18 \times 0.14$ mm; monoclinic; $C2/c$; $a = 25.7275(13)$, $b = 13.3117(7)$, $c = 23.8412(12)$ Å; $\beta = 109.791(3)°$; $V = 7.682.8(7)$ Å^3 ($Z = 4$); $\rho_{\text{calcd}} = 1.485$ Mg m^{-3}; $2\theta_{\max} = 33.21°$; $-39 < h < 28$, $-21 < k < 21$, $-22 < l < 22$; $\lambda = 0.71073$ Å; $T = 100(2)$ K; no. reflections = 65856; no. independent reflections = 14579 (R(int) = 0.0400); restraints/−parameters = 0/662; full-matrix least-squares refinement on F^2; semiempirical absorption correction from equivalents; $\mu = 0.148$ mm^{-1}; final R indices ($I > 2\sigma(I)$) are $R_1 = 0.0503$ and $wR_2 = 0.1215$; largest diff. peak and hole = 0.502 and −0.319 Å^{-3}. CCDC-629531 contains the supplementary crystallographic data for C_2-p^{11}-$C_{74}(CF_3)_{12}$. These data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data request/cif.

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[23] Crystals of C_2-p^{11}-(C_{78}-D_{3h}(5))(CF_3)$_{12}$·n(p-xylene) were grown in Berlin by slow evaporation of a saturated p-xylene solution: $0.5 \times 0.1 \times 0.05$ mm; triclinic; space group $P\bar{1}$ (No. 2); $a = 14.305(7)$, $b = 15.264(8)$, $c = 20.733(9)$ Å; $\alpha = 97.23(2)°$, $\beta = 98.96(2)°$, $\gamma = 94.32(1)°$, $V = 4415.7(4)$ Å^3, $Z = 2$; $2\theta_{\max} = 45.9°$; $-15 < h < 15$, $-16 < k < 16$, $-22 < l < 22$; $\lambda = 0.71073$ Å; $T = 153(2)$ K; no. reflections = 33961; no. independent reflections = 12151; restraints/-parameters = 0/1.136; full-matrix least-squares refinement on F^2; semiempirical absorption correction from equivalents; $\mu = 0.131$ mm^{-1} (transmission min/max = 0.88/1.00); final R indices ($F_o > 4\sigma(F_o)$) are $R_1 = 0.0734$ and $wR_2 = 0.204$ (R_1 (all data) = 0.100 and wR_2 (all data) = 0.218). CCDC-629280 (C_2-p^{11}-$C_{78}(CF_3)_{12}$) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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