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1,4,7,11,18,21,24,31,35,39,51,58,61,64-Tetradecakis(trifluoromethyl)-1,4,7,11,-18,21,24,31,35,39,51,58,61,64-tetradecahydro(C_{70}–D_{5h})[5,6]fullerene *p*-xylene trisolvate

Natalia B. Shustova,[a] Dimitry V. Peryshkov,[a] Ivan E. Kareev,[b] Olga V. Boltalina[a] and Steven H. Strauss[a]*

[a]Department of Chemistry, Colorado State University, Fort Collins, CO 80523, USA, and [b]Institute of Problems of Chemical Physics, Russian Academy of Sciences, Chernogolovka 142432, Russian Federation
Correspondence e-mail: steven.strauss@colostate.edu

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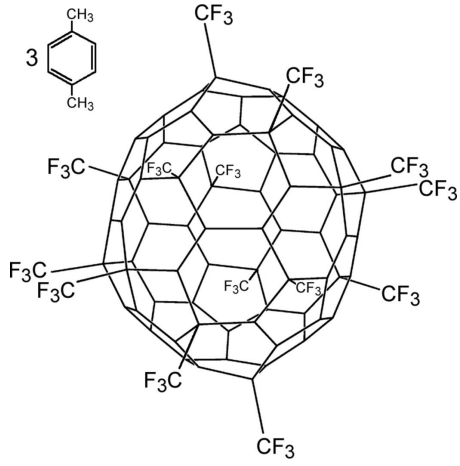
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.055; wR factor = 0.161; data-to-parameter ratio = 13.7.

The title compound, $C_{84}F_{42}\cdot 3C_8H_{10}$, features one of five isomers of $C_{70}(CF_3)_{14}$ that have been isolated. The fullerene molecule, which has crystallographic C_2 symmetry, consists of an idealized D_{5h}–C_{70} core with the 14 CF_3 groups arranged in an asymmetric fashion on a *para–para–para–para–para–para–para–meta–para* (p^7mp) ribbon and a *para–meta–para* (*pmp*) ribbon of edge-sharing $C_6(CF_3)_2$ hexagons such that the two ribbons connect to one another, forming two 1,3-$C_5(CF_3)_2$ pentagons. There are no cage Csp^3—Csp^3 bonds. There are intramolecular F···F contacts between pairs of CF_3 groups on the same hexagon that range from 2.560 (3) to 2.876 (3) Å.



Related literature

For related literature, see: Avdoshenko *et al.* (2006); Goryunkov *et al.* (2006); Ignat'eva *et al.* (2006); Kareev *et al.* (2005, 2006*a*,*b*); Olmstead *et al.* (2003); Popov *et al.* (2007); Powell *et al.* (2002).

Experimental

Crystal data

$C_{84}F_{42}\cdot 3C_8H_{10}$
M_r = 2125.32
Monoclinic, $C2/c$
a = 25.4423 (16) Å
b = 14.1495 (7) Å
c = 22.6519 (11) Å
β = 107.070 (5)°
V = 7795.4 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 100 (1) K
0.40 × 0.23 × 0.09 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*, Bruker, 2000)
T_{min} = 0.933, T_{max} = 0.984
130931 measured reflections
9305 independent reflections
6651 reflections with $I > 2\sigma(I)$
R_{int} = 0.066

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.161
S = 1.03
9305 reflections
679 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); 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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2073).

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supplementary materials

Acta Cryst. (2007). E**63**, o3928-o3929 [**doi:10.1107/S1600536807040755**]

1,4,7,11,18,21,24,31,35,39,51,58,61,64-Tetradecakis(trifluoromethyl)-1,4,7,11,18,21,24,31,35,39,51,58,61,64-tetradecahydro(C_{70}-D_{5h})[5,6]fullerene *p*-xylene trisolvate

N. B. Shustova, D. V. Peryshkov, I. E. Kareev, O. V. Boltalina and S. H. Strauss

Comment

Recently reported high-temperature reactions of C_{70} with CF_3I have yielded twenty-five $C_{70}(CF_3)_n$ derivatives (n = 2–18), most with relatively stable addition patterns that are chiral as well as unprecedented in fullerene$(X)_n$ chemistry (Kareev *et al.*, 2005; Kareev *et al.*, 2006*a*; Kareev *et al.*, 2006*b*; Avdoshenko *et al.*, 2006; Goryunkov *et al.*, 2006; Ignat'eva *et al.*, 2006; Popov *et al.*, 2007). A member of the n = 14 set of five isomers, the title compound, (I), has been crystallized from *p*-xylene and we report its crystal structure here. A much lower-quality structure (C—C su's 0.015–0.019 Å, R_1 = 0.186, wR_2 = 0.41) of the same fullerene molecule as a hexane solvate has recently been reported (Goryunkov *et al.*, 2006).

The structure of (I), Figs. 1 and 2, comprises an idealized D_{5h} C_{70} core with fourteen sp^3 carbon atoms at positions 1, 4, 7, 11, 18, 21, 24, 31, 35, 39, 51, 58, 61, and 64 (Powell *et al.*, 2002), each of which is attached to a CF_3 group. The molecule has crystallographic C_2 symmetry; symmetry related atoms have the letter a after the atom number. The core sp^3 carbon atoms are not adjacent to one another. The CF_3 groups are arranged on a *para-para-para-para-para-para-para- meta-para* (p^7mp) ribbon and a *para-meta-para* (pmp) ribbon of edge-sharing $C_6(CF_3)_2$ hexagons such that the two ribbons connect to one another, forming two 1,3-$C_5(CF_3)_2$ pentagons (see Schlegel diagram in Fig. 2). The shared edges in each ribbon of hexagons are C(sp^3)-C(sp^2) bonds (*e.g.*, C16—C17, C4—C18, *etc.*), not C(sp^2)-C(sp^2) bonds. Thus, any pair of adjacent hexagons along the two ribbons have a common CF_3 group. As in all other published structures of fullerene$(CF_3)_n$ compounds, there are F···F intramolecular contacts between pairs of neighboring CF_3 groups that range from 2.560 (3) to 2.876 (3) Å.

The four shortest cage C—C bonds (two pairs) in (I) are C1—C6a/C1a—C6, at 1.347 (3) Å, and C3—C4/C3a—C4a, at 1.356 (3) Å. All four are significantly shorter than the shortest C—C bond in the most precise structure of empty C_{60} reported to date (C_{60}·Pt(octaethylporphyrin)), which is 1.379 (3) Å (Olmstead *et al.*, 2003). More importantly, the C1—C6a and C1a—C6 bonds are pentagon-hexagon junctions, and the shortest pent-hex junction in C_{60}·Pt(OEP) is 1.440 (3) Å (the longest pent-hex junction in C_{60}·Pt(OEP) is 1.461 (3) Å); OEP is octaethylporphyrin).

Experimental

The synthesis of (I) was carried out by heating C_{70} in a stream of CF_3I at 420 °C as previously described (Popov *et al.*, 2007). Crystals of the HPLC-purified compound were grown by slow evaporation of a saturated deuterochloroform solution.

Refinement

The maximum (0.52 e/Å^3) and minimum (−0.53 e/Å^3) residual electron density peaks were located 1.07 Å from F422 and 0.58 Å from F391. The H atoms were geometrically placed (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(methyl C)

Figures

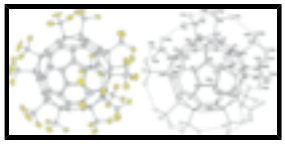

Fig. 1. (Left) The molecular structure of (I). Displacement ellipsoids are shown at the 50% probability level. (Right) The numbering scheme of (I). The crystallographic C_2 axis passes through the hexagon containing C29, C29*a*, C31, and C31*a* (symmetry related atoms have the letter a after the atom number).

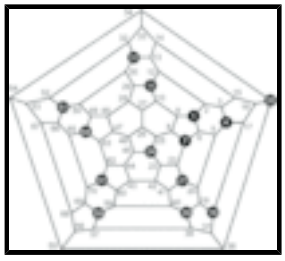

Fig. 2. Schlegel diagram of (I) showing the location of the CF_3 groups as black circles, the IUPAC lowest-locant numbers for the cage carbon atoms to which they are attached, and the ribbons of *meta*- and *para*-$C_6(CF_3)_2$ edge-sharing hexagons (*meta*-$C_6(CF_3)_2$ hexagons are indicated by the letter m).

1,4,7,11,18,21,24,31,35,39,51,58,61,64-Tetradecakis(trifluoromethyl)- 1,4,7,11,18,21,24,31,35,39,51,58,61,64-tetradecahydro(C_{70}—$D_{5\ h}$)[5,6]fullerene *p*-xylene trisolvate

Crystal data

$C_{84}F_{42}·3C_8H_{10}$	F_{000} = 4224
M_r = 2125.32	D_x = 1.811 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 25.4423 (16) Å	Cell parameters from 999 reflections
b = 14.1495 (7) Å	θ = 1.7–27.9º
c = 22.6519 (11) Å	μ = 0.18 mm^{-1}
β = 107.070 (5)º	*T* = 100 (1) K
V = 7795.4 (7) Å^3	Rhombic, orange
Z = 4	0.40 × 0.23 × 0.09 mm

Data collection

Bruker Kappa APEX II diffractometer	9305 independent reflections
Radiation source: fine-focus sealed tube	6651 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.066
T = 100(1) K	θ_{max} = 27.9º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan	*h* = −33→33

(SADABS, Bruker, 2000)

T_{min} = 0.933, T_{max} = 0.984

130931 measured reflections

k = −18→18

l = −29→29

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.055

$wR(F^2)$ = 0.161

S = 1.04

9305 reflections

679 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0719P)^2 + 26.7468P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.53 e Å^{-3}

$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.08268 (11)	0.48440 (17)	0.79209 (11)	0.0160 (5)
C2	−0.03691 (10)	0.46569 (17)	0.85024 (11)	0.0155 (5)
C3	0.01826 (10)	0.46278 (16)	0.83587 (11)	0.0154 (5)
C4	0.02365 (10)	0.44834 (16)	0.77873 (11)	0.0147 (5)
C5	0.08103 (11)	0.45187 (17)	0.77182 (11)	0.0160 (5)
C6	0.12112 (10)	0.55193 (17)	0.71344 (11)	0.0160 (5)
C7	−0.15208 (10)	0.74444 (18)	0.75467 (11)	0.0169 (5)
C8	−0.13424 (10)	0.71890 (17)	0.81628 (11)	0.0166 (5)
C9	−0.12683 (10)	0.61496 (17)	0.83788 (11)	0.0163 (5)
C10	−0.07032 (10)	0.61925 (18)	0.88728 (11)	0.0157 (5)
C11	−0.02992 (11)	0.55333 (17)	0.89099 (11)	0.0152 (5)
C12	0.02623 (10)	0.57981 (17)	0.91181 (10)	0.0147 (5)
C13	0.06281 (11)	0.51229 (17)	0.88946 (11)	0.0171 (5)
C14	0.10346 (10)	0.56652 (17)	0.86427 (11)	0.0145 (5)
C15	0.11537 (10)	0.52832 (17)	0.81343 (11)	0.0155 (5)

C16	0.14151 (10)	0.58187 (18)	0.77804 (11)	0.0166 (5)
C17	0.15683 (10)	0.67436 (18)	0.79395 (11)	0.0166 (5)
C18	−0.07696 (11)	0.85962 (17)	0.83726 (11)	0.0170 (5)
C19	−0.09497 (11)	0.77643 (18)	0.85807 (11)	0.0166 (5)
C20	−0.05573 (10)	0.71536 (17)	0.90126 (10)	0.0154 (5)
C21	−0.00067 (11)	0.74112 (17)	0.92113 (10)	0.0157 (5)
C22	0.04088 (10)	0.67163 (17)	0.92801 (10)	0.0152 (5)
C23	0.09401 (10)	0.71747 (17)	0.92343 (11)	0.0157 (5)
C24	0.11795 (10)	0.66342 (17)	0.87978 (11)	0.0148 (5)
C25	0.14403 (10)	0.71565 (17)	0.84463 (11)	0.0157 (5)
C26	0.13972 (11)	0.82482 (17)	0.83513 (11)	0.0167 (5)
C27	0.13535 (10)	0.83146 (17)	0.76627 (11)	0.0164 (5)
C28	0.09857 (11)	0.88846 (17)	0.72555 (11)	0.0169 (5)
C29	0.05705 (11)	0.95174 (17)	0.74390 (12)	0.0177 (5)
C30	0.04806 (11)	0.91925 (17)	0.80457 (11)	0.0168 (5)
C31	−0.00411 (11)	0.93096 (16)	0.81014 (11)	0.0176 (5)
C32	−0.01942 (11)	0.88503 (16)	0.85809 (11)	0.0162 (5)
C33	0.01818 (11)	0.82719 (17)	0.89960 (11)	0.0160 (5)
C34	0.07156 (11)	0.81482 (17)	0.89430 (11)	0.0164 (5)
C35	0.08718 (10)	0.85996 (17)	0.84767 (11)	0.0158 (5)
C36	−0.04668 (12)	0.37394 (19)	0.88469 (12)	0.0224 (6)
C37	0.10842 (12)	0.35315 (18)	0.78534 (12)	0.0211 (5)
C38	−0.17389 (11)	0.58456 (19)	0.86314 (12)	0.0216 (5)
C39	0.09405 (11)	0.44407 (19)	0.94399 (12)	0.0214 (6)
C40	0.13581 (11)	0.72325 (18)	0.98796 (11)	0.0194 (5)
C41	0.19157 (11)	0.87962 (19)	0.87432 (12)	0.0209 (5)
C42	0.07547 (12)	1.05667 (18)	0.74822 (12)	0.0223 (6)
C43	0.21133 (13)	−0.0831 (3)	0.15373 (16)	0.0399 (8)
C44	0.22565 (14)	−0.0842 (3)	0.09892 (17)	0.0431 (9)
H44	0.2274	−0.1419	0.0798	0.052*
C45	0.23724 (14)	−0.0025 (3)	0.07215 (18)	0.0452 (9)
H45	0.2460	−0.0059	0.0351	0.054*
C46	0.23610 (15)	0.0854 (3)	0.0997 (2)	0.0483 (9)
C47	0.22347 (15)	0.0864 (3)	0.1556 (2)	0.0505 (10)
H47	0.2231	0.1437	0.1756	0.061*
C48	0.21147 (15)	0.0043 (3)	0.18204 (18)	0.0452 (9)
H48	0.2033	0.0075	0.2194	0.054*
C49	0.19740 (16)	−0.1722 (3)	0.18156 (19)	0.0495 (10)
H49A	0.1602	−0.1901	0.1605	0.074*
H49B	0.2011	−0.1619	0.2245	0.074*
H49C	0.2219	−0.2217	0.1775	0.074*
C50	0.24732 (19)	0.1753 (3)	0.0697 (2)	0.0653 (13)
H50A	0.2131	0.2028	0.0460	0.098*
H50B	0.2694	0.1613	0.0430	0.098*
H50C	0.2665	0.2191	0.1011	0.098*
C51	0.04232 (15)	0.0553 (2)	−0.01127 (15)	0.0338 (7)
C52	0.05432 (14)	−0.0201 (2)	0.03007 (14)	0.0339 (7)
H52	0.0908	−0.0345	0.0507	0.041*
C53	0.01247 (15)	−0.0740 (2)	0.04073 (14)	0.0342 (7)

H53	0.0215	−0.1241	0.0685	0.041*
C54	0.08786 (19)	0.1138 (3)	−0.0231 (2)	0.0563 (11)
H54A	0.0777	0.1322	−0.0658	0.084*
H54B	0.1210	0.0771	−0.0136	0.084*
H54C	0.0938	0.1692	0.0024	0.084*
F361	−0.10036 (7)	0.35690 (12)	0.87384 (8)	0.0321 (4)
F362	−0.02505 (8)	0.38325 (12)	0.94590 (7)	0.0316 (4)
F363	−0.02429 (7)	0.29735 (11)	0.86770 (8)	0.0287 (4)
F371	0.11667 (8)	0.32738 (12)	0.84394 (8)	0.0348 (4)
F372	0.07692 (8)	0.28743 (11)	0.74981 (8)	0.0325 (4)
F373	0.15695 (7)	0.35158 (12)	0.77421 (9)	0.0330 (4)
F381	−0.16901 (7)	0.49526 (11)	0.88247 (7)	0.0266 (4)
F382	−0.17586 (7)	0.63905 (12)	0.91103 (8)	0.0291 (4)
F383	−0.22224 (7)	0.59329 (13)	0.81948 (8)	0.0302 (4)
F391	0.07719 (8)	0.35491 (12)	0.93486 (8)	0.0322 (4)
F392	0.14813 (7)	0.44451 (13)	0.95243 (8)	0.0316 (4)
F393	0.08619 (7)	0.47138 (12)	0.99746 (7)	0.0284 (4)
F401	0.14444 (7)	0.63742 (12)	1.01375 (7)	0.0278 (4)
F402	0.18459 (7)	0.75489 (14)	0.98599 (7)	0.0337 (4)
F403	0.11813 (7)	0.77886 (12)	1.02572 (7)	0.0266 (4)
F411	0.23546 (8)	0.82497 (14)	0.88972 (11)	0.0487 (6)
F412	0.20312 (9)	0.95185 (15)	0.84387 (9)	0.0495 (6)
F413	0.18514 (9)	0.91388 (17)	0.92594 (9)	0.0493 (6)
F421	0.05096 (11)	1.10843 (13)	0.78088 (12)	0.0590 (7)
F422	0.12873 (8)	1.06442 (13)	0.77648 (11)	0.0484 (6)
F423	0.06605 (9)	1.09499 (12)	0.69320 (8)	0.0439 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0224 (13)	0.0140 (11)	0.0110 (11)	−0.0045 (9)	0.0042 (10)	−0.0001 (9)
C2	0.0215 (13)	0.0143 (11)	0.0110 (11)	−0.0005 (9)	0.0053 (10)	−0.0005 (9)
C3	0.0228 (13)	0.0099 (10)	0.0128 (11)	0.0008 (9)	0.0041 (10)	0.0033 (9)
C4	0.0228 (13)	0.0079 (10)	0.0131 (11)	0.0004 (9)	0.0051 (10)	0.0016 (9)
C5	0.0223 (13)	0.0145 (11)	0.0109 (11)	0.0018 (9)	0.0044 (10)	−0.0002 (9)
C6	0.0201 (13)	0.0156 (11)	0.0128 (11)	0.0047 (9)	0.0056 (10)	0.0000 (9)
C7	0.0173 (12)	0.0195 (12)	0.0147 (12)	0.0039 (9)	0.0060 (10)	0.0010 (10)
C8	0.0184 (12)	0.0176 (12)	0.0153 (12)	0.0032 (9)	0.0074 (10)	0.0017 (9)
C9	0.0204 (12)	0.0170 (12)	0.0124 (11)	−0.0001 (9)	0.0063 (10)	0.0005 (9)
C10	0.0208 (13)	0.0187 (12)	0.0085 (11)	−0.0019 (9)	0.0057 (9)	0.0016 (9)
C11	0.0242 (13)	0.0135 (11)	0.0083 (11)	−0.0007 (9)	0.0055 (9)	0.0027 (9)
C12	0.0221 (13)	0.0152 (11)	0.0064 (10)	0.0014 (9)	0.0037 (9)	0.0027 (9)
C13	0.0227 (13)	0.0155 (11)	0.0122 (11)	0.0027 (10)	0.0038 (10)	0.0003 (9)
C14	0.0167 (12)	0.0154 (11)	0.0092 (11)	0.0016 (9)	0.0004 (9)	0.0032 (9)
C15	0.0179 (12)	0.0142 (11)	0.0125 (11)	0.0022 (9)	0.0013 (9)	0.0010 (9)
C16	0.0192 (12)	0.0183 (12)	0.0107 (11)	0.0034 (9)	0.0020 (9)	0.0000 (9)
C17	0.0176 (12)	0.0195 (12)	0.0123 (11)	−0.0006 (9)	0.0038 (9)	−0.0002 (9)
C18	0.0243 (13)	0.0143 (11)	0.0132 (12)	0.0043 (10)	0.0067 (10)	−0.0029 (9)

C19	0.0211 (13)	0.0186 (12)	0.0121 (11)	0.0037 (10)	0.0080 (10)	−0.0012 (9)
C20	0.0234 (13)	0.0179 (12)	0.0060 (10)	0.0020 (10)	0.0060 (9)	0.0005 (9)
C21	0.0277 (14)	0.0157 (11)	0.0041 (10)	0.0008 (10)	0.0052 (9)	−0.0021 (8)
C22	0.0207 (12)	0.0189 (12)	0.0055 (10)	0.0002 (9)	0.0031 (9)	0.0010 (9)
C23	0.0218 (13)	0.0156 (11)	0.0076 (11)	0.0001 (9)	0.0008 (9)	−0.0020 (9)
C24	0.0163 (12)	0.0178 (11)	0.0080 (11)	0.0015 (9)	−0.0004 (9)	−0.0002 (9)
C25	0.0166 (12)	0.0167 (11)	0.0118 (11)	−0.0001 (9)	0.0012 (9)	−0.0015 (9)
C26	0.0213 (13)	0.0150 (11)	0.0130 (11)	−0.0033 (9)	0.0039 (10)	−0.0006 (9)
C27	0.0195 (12)	0.0160 (11)	0.0140 (12)	−0.0059 (9)	0.0055 (10)	−0.0015 (9)
C28	0.0238 (13)	0.0121 (11)	0.0155 (12)	−0.0065 (9)	0.0071 (10)	−0.0009 (9)
C29	0.0263 (14)	0.0104 (11)	0.0161 (12)	−0.0034 (9)	0.0057 (10)	0.0002 (9)
C30	0.0272 (13)	0.0105 (10)	0.0120 (11)	−0.0023 (9)	0.0048 (10)	−0.0028 (9)
C31	0.0295 (14)	0.0090 (11)	0.0132 (11)	−0.0001 (9)	0.0047 (10)	−0.0031 (9)
C32	0.0270 (13)	0.0106 (10)	0.0128 (11)	0.0016 (9)	0.0084 (10)	−0.0040 (9)
C33	0.0258 (13)	0.0135 (11)	0.0084 (11)	−0.0005 (9)	0.0046 (9)	−0.0037 (9)
C34	0.0245 (13)	0.0139 (11)	0.0090 (11)	−0.0015 (9)	0.0023 (10)	−0.0047 (9)
C35	0.0235 (13)	0.0115 (11)	0.0116 (11)	−0.0035 (9)	0.0041 (10)	−0.0041 (9)
C36	0.0321 (15)	0.0191 (13)	0.0156 (12)	−0.0020 (11)	0.0064 (11)	0.0012 (10)
C37	0.0288 (14)	0.0174 (12)	0.0164 (12)	0.0039 (10)	0.0057 (11)	0.0010 (10)
C38	0.0238 (14)	0.0245 (13)	0.0165 (12)	0.0005 (11)	0.0060 (10)	0.0021 (10)
C39	0.0262 (14)	0.0230 (13)	0.0144 (12)	0.0020 (10)	0.0047 (11)	0.0030 (10)
C40	0.0246 (14)	0.0213 (13)	0.0113 (11)	−0.0010 (10)	0.0037 (10)	−0.0031 (10)
C41	0.0261 (14)	0.0202 (13)	0.0156 (12)	−0.0041 (10)	0.0050 (11)	−0.0022 (10)
C42	0.0353 (16)	0.0144 (12)	0.0177 (13)	−0.0028 (10)	0.0086 (11)	−0.0003 (10)
C43	0.0211 (15)	0.056 (2)	0.0365 (18)	0.0034 (14)	−0.0006 (13)	−0.0158 (16)
C44	0.0291 (17)	0.051 (2)	0.043 (2)	−0.0020 (15)	0.0020 (15)	−0.0232 (17)
C45	0.0315 (18)	0.058 (2)	0.040 (2)	0.0035 (16)	0.0015 (15)	−0.0123 (17)
C46	0.0283 (18)	0.052 (2)	0.058 (2)	0.0107 (16)	0.0031 (16)	−0.0054 (19)
C47	0.0340 (19)	0.051 (2)	0.063 (3)	0.0080 (16)	0.0097 (18)	−0.023 (2)
C48	0.0332 (19)	0.055 (2)	0.046 (2)	0.0074 (16)	0.0085 (16)	−0.0174 (18)
C49	0.0344 (19)	0.060 (2)	0.053 (2)	−0.0050 (17)	0.0109 (17)	−0.0171 (19)
C50	0.049 (2)	0.058 (3)	0.086 (3)	0.017 (2)	0.015 (2)	0.007 (2)
C51	0.048 (2)	0.0254 (15)	0.0297 (16)	0.0011 (13)	0.0140 (14)	−0.0090 (12)
C52	0.0391 (18)	0.0298 (16)	0.0267 (16)	0.0115 (13)	0.0003 (13)	−0.0101 (12)
C53	0.057 (2)	0.0219 (14)	0.0213 (14)	0.0087 (14)	0.0083 (14)	−0.0019 (11)
C54	0.067 (3)	0.044 (2)	0.069 (3)	−0.0063 (19)	0.037 (2)	−0.009 (2)
F361	0.0343 (10)	0.0280 (9)	0.0369 (10)	−0.0051 (7)	0.0151 (8)	0.0093 (7)
F362	0.0511 (11)	0.0265 (9)	0.0158 (8)	−0.0014 (8)	0.0077 (7)	0.0056 (7)
F363	0.0446 (10)	0.0162 (7)	0.0276 (9)	0.0019 (7)	0.0142 (8)	0.0027 (6)
F371	0.0533 (12)	0.0298 (9)	0.0206 (8)	0.0159 (8)	0.0095 (8)	0.0085 (7)
F372	0.0421 (10)	0.0163 (8)	0.0334 (10)	0.0005 (7)	0.0024 (8)	−0.0031 (7)
F373	0.0310 (10)	0.0277 (9)	0.0425 (11)	0.0119 (7)	0.0140 (8)	0.0057 (8)
F381	0.0309 (9)	0.0241 (8)	0.0273 (9)	−0.0027 (7)	0.0120 (7)	0.0070 (7)
F382	0.0342 (9)	0.0347 (9)	0.0243 (9)	0.0018 (7)	0.0178 (7)	−0.0009 (7)
F383	0.0211 (8)	0.0432 (10)	0.0247 (9)	−0.0023 (7)	0.0043 (7)	0.0092 (7)
F391	0.0465 (11)	0.0221 (8)	0.0243 (9)	0.0002 (7)	0.0047 (8)	0.0056 (7)
F392	0.0274 (9)	0.0383 (10)	0.0278 (9)	0.0090 (7)	0.0062 (7)	0.0111 (7)
F393	0.0391 (10)	0.0326 (9)	0.0126 (7)	0.0083 (7)	0.0060 (7)	0.0034 (6)
F401	0.0351 (9)	0.0268 (8)	0.0140 (8)	0.0043 (7)	−0.0044 (7)	0.0035 (6)

F402	0.0257 (9)	0.0562 (12)	0.0173 (8)	−0.0161 (8)	0.0033 (7)	−0.0080 (8)
F403	0.0341 (9)	0.0311 (9)	0.0115 (7)	0.0038 (7)	0.0018 (6)	−0.0074 (6)
F411	0.0236 (10)	0.0376 (11)	0.0690 (15)	0.0009 (8)	−0.0112 (9)	−0.0171 (10)
F412	0.0535 (13)	0.0463 (12)	0.0342 (11)	−0.0352 (10)	−0.0097 (9)	0.0147 (9)
F413	0.0475 (12)	0.0731 (15)	0.0312 (10)	−0.0330 (11)	0.0174 (9)	−0.0349 (10)
F421	0.1002 (19)	0.0159 (9)	0.0904 (18)	−0.0115 (10)	0.0739 (16)	−0.0133 (10)
F422	0.0388 (11)	0.0223 (9)	0.0680 (14)	−0.0106 (8)	−0.0094 (10)	−0.0040 (9)
F423	0.0774 (15)	0.0232 (9)	0.0219 (9)	−0.0229 (9)	0.0001 (9)	0.0078 (7)

Geometric parameters (Å, °)

C1—C6^{i}	1.346 (4)	C29—C42	1.551 (3)
C1—C2	1.504 (3)	C29—C31^{i}	1.559 (4)
C1—C5^{i}	1.532 (3)	C30—C31	1.380 (4)
C2—C11	1.525 (3)	C30—C35	1.441 (3)
C2—C3	1.532 (4)	C31—C32	1.415 (4)
C2—C36	1.572 (3)	C31—C29^{i}	1.559 (4)
C3—C4	1.356 (3)	C32—C33	1.393 (3)
C3—C13	1.562 (3)	C33—C34	1.409 (4)
C4—C4^{i}	1.491 (5)	C34—C35	1.388 (4)
C4—C5	1.514 (4)	C36—F363	1.333 (3)
C5—C15	1.529 (3)	C36—F361	1.337 (3)
C5—C1^{i}	1.532 (3)	C36—F362	1.340 (3)
C5—C37	1.551 (3)	C37—F373	1.331 (3)
C6—C1^{i}	1.346 (4)	C37—F371	1.333 (3)
C6—C16	1.464 (3)	C37—F372	1.333 (3)
C6—C9^{i}	1.506 (3)	C38—F381	1.331 (3)
C7—C8	1.383 (3)	C38—F383	1.339 (3)
C7—C27^{i}	1.429 (4)	C38—F382	1.344 (3)
C7—C17^{i}	1.460 (3)	C39—F391	1.329 (3)
C8—C19	1.415 (4)	C39—F392	1.332 (3)
C8—C9	1.544 (3)	C39—F393	1.341 (3)
C9—C6^{i}	1.506 (3)	C40—F402	1.332 (3)
C9—C38	1.532 (4)	C40—F403	1.334 (3)
C9—C10	1.542 (3)	C40—F401	1.338 (3)
C10—C11	1.372 (4)	C41—F412	1.314 (3)
C10—C20	1.421 (3)	C41—F411	1.318 (3)
C11—C12	1.417 (4)	C41—F413	1.319 (3)
C12—C22	1.372 (3)	C42—F423	1.315 (3)
C12—C13	1.521 (3)	C42—F421	1.321 (3)
C13—C14	1.526 (4)	C42—F422	1.322 (3)
C13—C39	1.585 (3)	C43—C44	1.393 (5)
C14—C15	1.384 (3)	C43—C48	1.393 (5)
C14—C24	1.436 (3)	C43—C49	1.498 (6)
C15—C16	1.404 (4)	C44—C45	1.378 (6)
C16—C17	1.383 (4)	C44—H44	0.9300
C17—C25	1.409 (3)	C45—C46	1.396 (5)

C17—C7^{i}	1.460 (3)	C45—H45	0.9300
C18—C19	1.394 (4)	C46—C47	1.394 (6)
C18—C28^{i}	1.426 (3)	C46—C50	1.509 (6)
C18—C32	1.445 (4)	C47—C48	1.380 (6)
C19—C20	1.459 (3)	C47—H47	0.9300
C20—C21	1.389 (4)	C48—H48	0.9300
C21—C22	1.418 (3)	C49—H49A	0.9600
C21—C33	1.445 (3)	C49—H49B	0.9600
C22—C23	1.530 (4)	C49—H49C	0.9600
C23—C24	1.513 (3)	C50—H50A	0.9600
C23—C40	1.537 (3)	C50—H50B	0.9600
C23—C34	1.561 (3)	C50—H50C	0.9600
C24—C25	1.389 (4)	C51—C53^{ii}	1.383 (5)
C25—C26	1.559 (3)	C51—C52	1.393 (5)
C26—C35	1.529 (4)	C51—C54	1.511 (5)
C26—C27	1.534 (3)	C52—C53	1.387 (5)
C26—C41	1.563 (4)	C52—H52	0.9300
C27—C28	1.368 (4)	C53—C51^{ii}	1.383 (5)
C27—C7^{i}	1.429 (4)	C53—H53	0.9300
C28—C18^{i}	1.426 (3)	C54—H54A	0.9600
C28—C29	1.532 (4)	C54—H54B	0.9600
C29—C30	1.529 (3)	C54—H54C	0.9600
C6^{i}—C1—C2	124.1 (2)	C42—C29—C31^{i}	113.9 (2)
C6^{i}—C1—C5^{i}	110.2 (2)	C31—C30—C35	120.0 (2)
C2—C1—C5^{i}	123.1 (2)	C31—C30—C29	116.6 (2)
C1—C2—C11	108.1 (2)	C35—C30—C29	121.9 (2)
C1—C2—C3	109.9 (2)	C30—C31—C32	120.3 (2)
C11—C2—C3	101.5 (2)	C30—C31—C29^{i}	125.7 (2)
C1—C2—C36	112.7 (2)	C32—C31—C29^{i}	108.7 (2)
C11—C2—C36	112.2 (2)	C33—C32—C31	119.9 (2)
C3—C2—C36	111.8 (2)	C33—C32—C18	120.2 (2)
C4—C3—C2	124.2 (2)	C31—C32—C18	110.1 (2)
C4—C3—C13	123.7 (2)	C32—C33—C34	120.2 (2)
C2—C3—C13	108.9 (2)	C32—C33—C21	119.5 (2)
C3—C4—C4^{i}	123.5 (3)	C34—C33—C21	110.3 (2)
C3—C4—C5	117.7 (2)	C35—C34—C33	120.4 (2)
C4^{i}—C4—C5	117.7 (3)	C35—C34—C23	125.2 (2)
C4—C5—C15	110.7 (2)	C33—C34—C23	108.4 (2)
C4—C5—C1^{i}	113.7 (2)	C34—C35—C30	119.2 (2)
C15—C5—C1^{i}	101.6 (2)	C34—C35—C26	116.9 (2)
C4—C5—C37	109.9 (2)	C30—C35—C26	122.1 (2)
C15—C5—C37	111.9 (2)	F363—C36—F361	107.5 (2)
C1^{i}—C5—C37	108.8 (2)	F363—C36—F362	107.4 (2)
C1^{i}—C6—C16	109.4 (2)	F361—C36—F362	107.2 (2)
C1^{i}—C6—C9^{i}	125.5 (2)	F363—C36—C2	112.7 (2)

C16—C6—C9^{i}	120.9 (2)	F361—C36—C2	111.1 (2)
C8—C7—C27^{i}	121.2 (2)	F362—C36—C2	110.9 (2)
C8—C7—C17^{i}	120.9 (2)	F373—C37—F371	107.5 (2)
C27^{i}—C7—C17^{i}	107.5 (2)	F373—C37—F372	107.5 (2)
C7—C8—C19	118.9 (2)	F371—C37—F372	107.5 (2)
C7—C8—C9	122.8 (2)	F373—C37—C5	111.5 (2)
C19—C8—C9	109.9 (2)	F371—C37—C5	111.9 (2)
C6^{i}—C9—C38	112.7 (2)	F372—C37—C5	110.7 (2)
C6^{i}—C9—C10	107.6 (2)	F381—C38—F383	107.9 (2)
C38—C9—C10	113.8 (2)	F381—C38—F382	107.5 (2)
C6^{i}—C9—C8	110.5 (2)	F383—C38—F382	107.4 (2)
C38—C9—C8	110.5 (2)	F381—C38—C9	112.4 (2)
C10—C9—C8	101.2 (2)	F383—C38—C9	110.3 (2)
C11—C10—C20	119.7 (2)	F382—C38—C9	111.0 (2)
C11—C10—C9	122.7 (2)	F391—C39—F392	107.4 (2)
C20—C10—C9	109.1 (2)	F391—C39—F393	106.7 (2)
C10—C11—C12	120.3 (2)	F392—C39—F393	106.9 (2)
C10—C11—C2	124.3 (2)	F391—C39—C13	113.1 (2)
C12—C11—C2	110.1 (2)	F392—C39—C13	111.7 (2)
C22—C12—C11	120.5 (2)	F393—C39—C13	110.8 (2)
C22—C12—C13	122.7 (2)	F402—C40—F403	108.4 (2)
C11—C12—C13	112.3 (2)	F402—C40—F401	106.5 (2)
C12—C13—C14	110.9 (2)	F403—C40—F401	107.4 (2)
C12—C13—C3	99.5 (2)	F402—C40—C23	112.2 (2)
C14—C13—C3	110.6 (2)	F403—C40—C23	111.8 (2)
C12—C13—C39	109.8 (2)	F401—C40—C23	110.3 (2)
C14—C13—C39	111.0 (2)	F412—C41—F411	107.0 (2)
C3—C13—C39	114.6 (2)	F412—C41—F413	106.5 (2)
C15—C14—C24	118.2 (2)	F411—C41—F413	107.3 (2)
C15—C14—C13	116.9 (2)	F412—C41—C26	111.7 (2)
C24—C14—C13	122.8 (2)	F411—C41—C26	111.6 (2)
C14—C15—C16	121.4 (2)	F413—C41—C26	112.5 (2)
C14—C15—C5	124.3 (2)	F423—C42—F421	108.5 (2)
C16—C15—C5	108.1 (2)	F423—C42—F422	107.5 (2)
C17—C16—C15	120.4 (2)	F421—C42—F422	105.6 (2)
C17—C16—C6	121.6 (2)	F423—C42—C29	111.5 (2)
C15—C16—C6	109.0 (2)	F421—C42—C29	112.5 (2)
C16—C17—C25	119.3 (2)	F422—C42—C29	111.0 (2)
C16—C17—C7^{i}	119.4 (2)	C44—C43—C48	117.1 (4)
C25—C17—C7^{i}	110.2 (2)	C44—C43—C49	121.4 (3)
C19—C18—C28^{i}	120.7 (2)	C48—C43—C49	121.5 (3)
C19—C18—C32	120.1 (2)	C45—C44—C43	121.9 (3)
C28^{i}—C18—C32	108.1 (2)	C45—C44—H44	119.1
C18—C19—C8	119.8 (2)	C43—C44—H44	119.1
C18—C19—C20	119.9 (2)	C44—C45—C46	121.1 (4)
C8—C19—C20	108.5 (2)	C44—C45—H45	119.5
C21—C20—C10	119.8 (2)	C46—C45—H45	119.5

C21—C20—C19	119.3 (2)	C47—C46—C45	117.1 (4)
C10—C20—C19	109.6 (2)	C47—C46—C50	121.7 (4)
C20—C21—C22	120.1 (2)	C45—C46—C50	121.2 (4)
C20—C21—C33	121.0 (2)	C48—C47—C46	121.7 (4)
C22—C21—C33	108.1 (2)	C48—C47—H47	119.2
C12—C22—C21	119.5 (2)	C46—C47—H47	119.2
C12—C22—C23	124.0 (2)	C47—C48—C43	121.2 (4)
C21—C22—C23	110.1 (2)	C47—C48—H48	119.4
C24—C23—C22	111.3 (2)	C43—C48—H48	119.4
C24—C23—C40	110.5 (2)	C43—C49—H49A	109.5
C22—C23—C40	109.7 (2)	C43—C49—H49B	109.5
C24—C23—C34	109.71 (19)	H49A—C49—H49B	109.5
C22—C23—C34	100.5 (2)	C43—C49—H49C	109.5
C40—C23—C34	114.9 (2)	H49A—C49—H49C	109.5
C25—C24—C14	119.9 (2)	H49B—C49—H49C	109.5
C25—C24—C23	117.2 (2)	C46—C50—H50A	109.5
C14—C24—C23	121.3 (2)	C46—C50—H50B	109.5
C24—C25—C17	120.7 (2)	H50A—C50—H50B	109.5
C24—C25—C26	125.5 (2)	C46—C50—H50C	109.5
C17—C25—C26	108.8 (2)	H50A—C50—H50C	109.5
C35—C26—C27	111.3 (2)	H50B—C50—H50C	109.5
C35—C26—C25	109.1 (2)	C53^{ii}—C51—C52	117.5 (3)
C27—C26—C25	100.50 (19)	C53^{ii}—C51—C54	121.8 (3)
C35—C26—C41	111.8 (2)	C52—C51—C54	120.7 (3)
C27—C26—C41	110.2 (2)	C53—C52—C51	120.7 (3)
C25—C26—C41	113.6 (2)	C53—C52—H52	119.7
C28—C27—C7^{i}	119.7 (2)	C51—C52—H52	119.7
C28—C27—C26	123.6 (2)	C51^{ii}—C53—C52	121.8 (3)
C7^{i}—C27—C26	110.2 (2)	C51^{ii}—C53—H53	119.1
C27—C28—C18^{i}	119.4 (2)	C52—C53—H53	119.1
C27—C28—C29	123.5 (2)	C51—C54—H54A	109.5
C18^{i}—C28—C29	110.1 (2)	C51—C54—H54B	109.5
C30—C29—C28	111.4 (2)	H54A—C54—H54B	109.5
C30—C29—C42	110.7 (2)	C51—C54—H54C	109.5
C28—C29—C42	110.9 (2)	H54A—C54—H54C	109.5
C30—C29—C31^{i}	109.0 (2)	H54B—C54—H54C	109.5
C28—C29—C31^{i}	100.51 (19)		

Symmetry codes: (i) $-x$, y, $-z+3/2$; (ii) $-x$, $-y$, $-z$.

Fig. 1

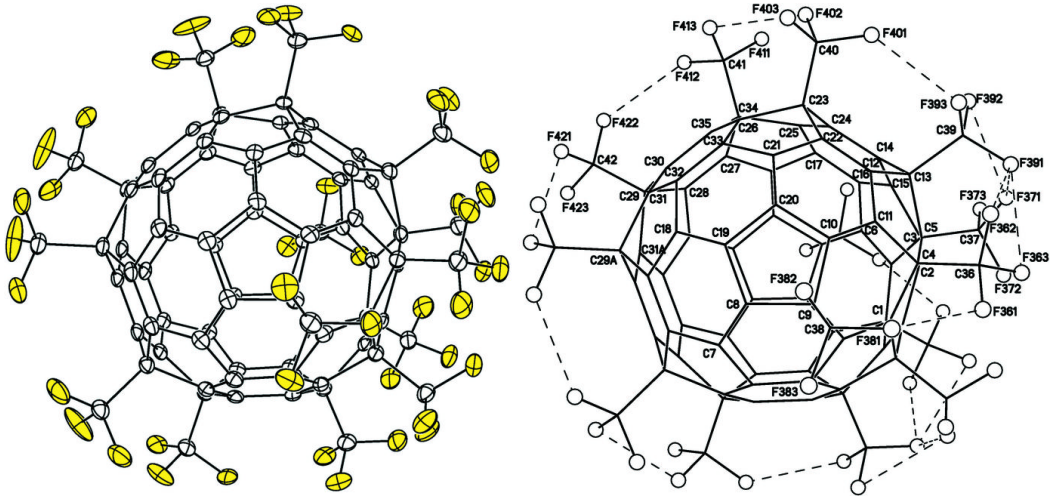
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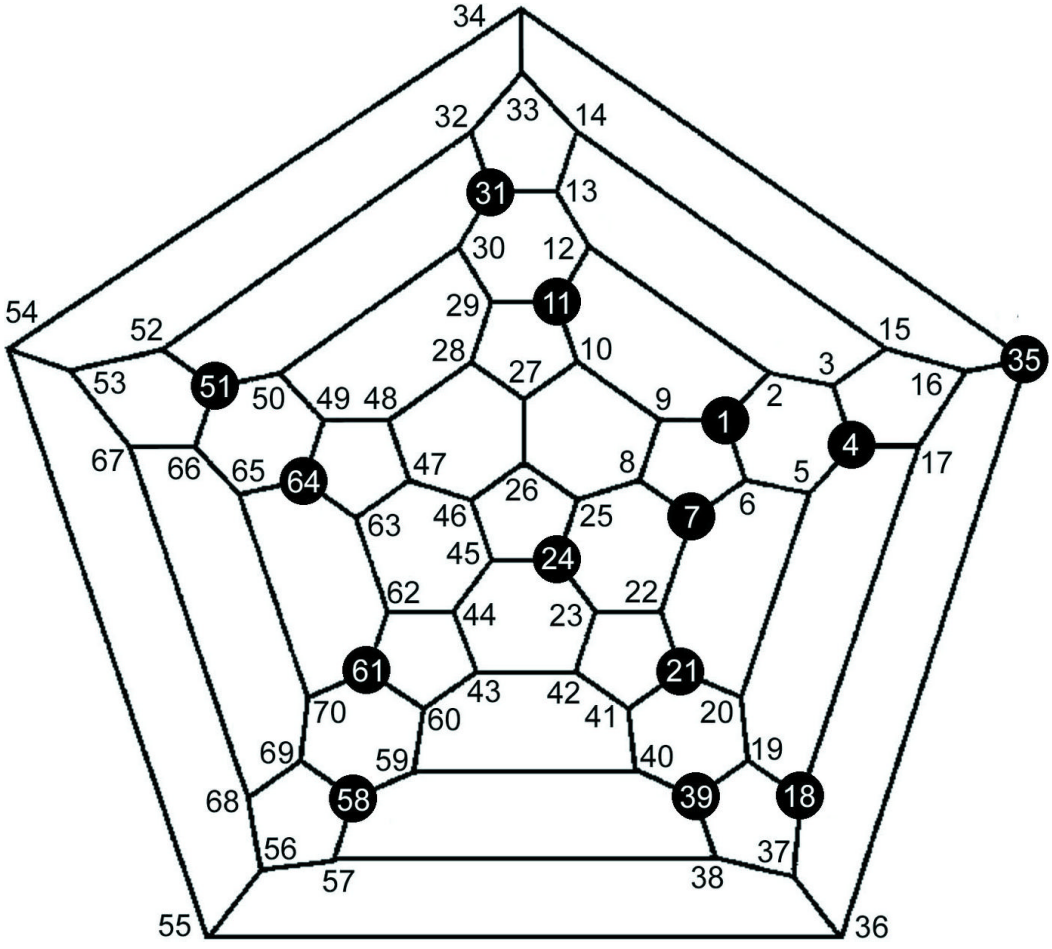
Fig. 2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