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C₆₀ 1,1,2,2-tetrachloroethylene tetrasolvate

C. Arunkumar,^a P. Bhyrappa^{a*} and B. Varghese^b

^aDepartment of Chemistry, Indian Institute of Technology Madras, Chennai 600 036, India, and ^bSophisticated Analytical Instrument Facility, Indian Institute of Technology Madras, Chennai 600 036, India

Correspondence e-mail: byra@iitm.ac.in

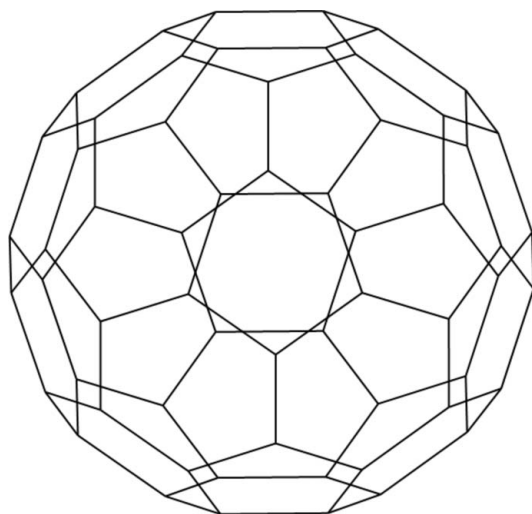
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.019$ Å; disorder in solvent or counterion; R factor = 0.105; wR factor = 0.265; data-to-parameter ratio = 9.6.

In the title complex, C₆₀·4C₂Cl₄, the C₆₀ molecule is located on an inversion centre and there are two tetrachloroethylene (TCE) molecules in the asymmetric unit. Both TCE molecules show positional disorder, with occupancy ratios of 0.75:0.25 and 0.56:0.44. Four fullerene C atoms form short contacts [3.208 (17) and 3.223 (17) Å] with the centres of the TCE double bonds, indicating that C₆₀-solvent interactions are largely π - π in nature.

Related literature

For related literature on inclusion compounds of C₆₀ with various guest molecules, see: Balch & Olmstead (1999) and references cited therein; Olmstead *et al.* (2000); Hardie *et al.* (2003); Bond (2003); Litvinov *et al.* (2003); Soldatov *et al.* (2001); Dodrick *et al.* (2005).



Experimental

Crystal data

C ₆₀ ·4C ₂ Cl ₄	$\gamma = 79.834$ (5)°
$M_r = 1383.88$	$V = 1199.6$ (10) Å ³
Triclinic, $P\bar{1}$	$Z = 1$
$a = 10.049$ (5) Å	Mo $K\alpha$ radiation
$b = 10.168$ (5) Å	$\mu = 0.97$ mm ⁻¹
$c = 13.412$ (5) Å	$T = 173$ (2) K
$\alpha = 70.484$ (5)°	$0.3 \times 0.2 \times 0.2$ mm
$\beta = 68.508$ (5)°	

Data collection

Bruker Kappa APEX2 diffractometer	11755 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 1999)	4047 independent reflections
$T_{\min} = 0.759$, $T_{\max} = 0.830$	3502 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.105$	420 parameters
$wR(F^2) = 0.265$	34 restraints
$S = 1.11$	$\Delta\rho_{\max} = 1.25$ e Å ⁻³
4047 reflections	$\Delta\rho_{\min} = -0.68$ e Å ⁻³

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2 and SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2113).

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supplementary materials

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C₆₀ 1,1,2,2-tetrachloroethylene tetrasolvate

C. Arunkumar, P. Bhyrappa and B. Varghese

Comment

C₆₀ is located around inversion centre and there are two symmetry independent 1,1,2,2-tetrachloroethylene (TCE) solvent molecules in the asymmetric unit. The *ORTEP* drawing of the title compound is shown in Fig. 1. In the C₆₀ molecule inter-pentagonal bond distances are in the range 1.3947 (17)–1.414 (15) Å while the intra-pentagonal bond distances are in the range 1.364 (19)–1.55 (2) Å. Such a variation in bond lengths is possibly due to the librational motion of the C₆₀ molecule in the crystal. Similar variations of bond lengths were observed in some other C₆₀ co-crystals (Dodrick *et al.*, 2005). The C₆₀ and TCE molecules show intermolecular π - π interactions (Fig. 2). The short contacts between the fullerene C2 atom and the centre of the ethylene C33A—C34A bond, and C22 atom and the centre of the ethylene C31A—C32A bond are of 3.208 (17) and 3.223 (17) Å, respectively. Additionally, short contacts are observed between Cl atoms of TCE and C₆₀ (C17 \cdots C14ⁱ 3.419 (14) Å, C15 \cdots C14 = 3.491 (4) Å; symmetry code: (i) $x, -1 + y, z$]. The solvent molecules and C₆₀ are located in alternating layers parallel to the (001) plane. The shortest C₆₀ \cdots C₆₀ contacts are: C27 \cdots C29ⁱ 3.690 (14) Å and C9 \cdots C11ⁱⁱ 3.447 (14) Å [symmetry code: (i) $2 - x, 1 - y, -z$, (ii) $1 - x, -y, -z$]

Experimental

C₆₀ sample was purchased from Sigma-Aldrich and used as received. The solvents for crystallization, 1,1,2,2-tetrachloroethylene (TCE) and methanol were of purity >99% and were purchased from E. Merck (Germany). Crystals of the C₆₀.4(CCl₄) were grown by diffusion of methanol into a solution of C₆₀ in TCE over a period of five days. Upon removal from the mother liquor the crystals were unstable and therefore were mounted at 0°C.

Refinement

The C₆₀ molecule was refined without any restraints. Both TCE molecules were disordered and showed two approximately perpendicular orientations with overlapping Cl atoms (the ratio of occupancies 0.75:0.25 and 0.56:0.44). Since one of the disordered TCE molecules showed occupancies very close to 0.75 and 0.25 these values were fixed at the final stages of the refinement. The restraints were imposed on C—C and C—Cl bond lengths of the TCE molecules and anisotropic displacement parameters of C31A, C32A and C34A atoms. The residual peak of 1.25 e Å⁻³ is located at 1.06 Å from the C5 atom of C₆₀ indicating that in addition to large librational motion the C₆₀ molecule can be also partially disordered. The high residual values, R1=0.105 and wR1=0.259 are most probably due to disorder of the TCE and C₆₀ molecules.

Figures

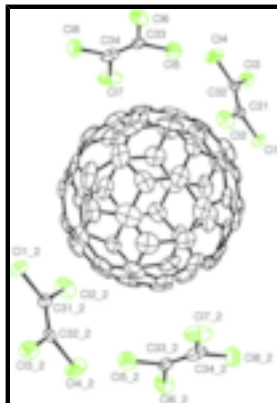


Fig. 1. ORTEP diagram of the title compound showing 50% probability displacement ellipsoids. Labels for C₆₀ were omitted. The TCE molecules in minor occupancies are not shown. Symmetry code for the atoms not from the asymmetric unit(₂): 2 - x, -y, -z).

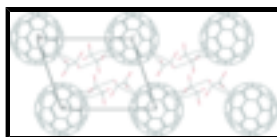


Fig. 2. View of the crystal packing in the title compound along the *b* axis.

(I)

Crystal data

C₆₀·4C₂Cl₄

M_r = 1383.88

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

a = 10.049 (5) Å

b = 10.168 (5) Å

c = 13.412 (5) Å

α = 70.484 (5)°

β = 68.508 (5)°

γ = 79.834 (5)°

V = 1199.6 (10) Å³

Z = 1

*F*₀₀₀ = 680

D_x = 1.916 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 9670 reflections

θ = 2.2–28.4°

μ = 0.97 mm⁻¹

T = 173 (2) K

Plate, brown

0.3 × 0.2 × 0.2 mm

Data collection

Bruker APEX2 Kappa
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 173(2) K

ω and ϕ scan

Absorption correction: multi-scan
(SADABS; Bruker, 1999)

*T*_{min} = 0.759, *T*_{max} = 0.830

11755 measured reflections

4047 independent reflections

3502 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.037

θ _{max} = 25.0°

θ _{min} = 2.1°

h = -11→11

k = -12→11

l = -15→15

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 20.5574P]$
$wR(F^2) = 0.265$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{\max} < 0.001$
4047 reflections	$\Delta\rho_{\max} = 1.25 \text{ e } \text{\AA}^{-3}$
420 parameters	$\Delta\rho_{\min} = -0.68 \text{ e } \text{\AA}^{-3}$
34 restraints	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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.7872 (12)	0.2020 (11)	0.1752 (10)	0.048 (3)	
C2	0.8649 (12)	0.1051 (13)	0.2458 (9)	0.050 (3)	
C3	0.8346 (14)	-0.0347 (13)	0.2881 (8)	0.053 (3)	
C4	0.7301 (14)	-0.0837 (17)	0.2621 (11)	0.069 (4)	
C5	0.6574 (12)	-0.0048 (16)	0.2046 (11)	0.069 (4)	
C6	0.6836 (10)	0.1484 (11)	0.1574 (9)	0.044 (3)	
C7	0.6674 (10)	0.1949 (11)	0.0509 (10)	0.043 (2)	
C8	0.6353 (9)	0.0806 (11)	0.0226 (9)	0.038 (2)	
C9	0.6293 (9)	-0.0449 (12)	0.1128 (11)	0.051 (3)	
C10	0.6808 (12)	-0.1691 (13)	0.0941 (14)	0.068 (4)	
C11	0.6954 (10)	0.0755 (11)	-0.0878 (9)	0.043 (3)	
C12	1.2500 (12)	0.0616 (14)	0.1058 (11)	0.055 (3)	
C13	1.2598 (12)	0.1767 (11)	0.0131 (12)	0.051 (3)	
C14	0.7609 (12)	-0.2663 (12)	0.1663 (12)	0.062 (4)	
C15	0.7816 (13)	-0.2274 (11)	0.2465 (9)	0.052 (3)	
C16	0.9231 (17)	-0.2585 (12)	0.2626 (9)	0.063 (4)	
C17	0.9541 (18)	-0.1434 (13)	0.2871 (8)	0.064 (4)	
C18	1.0919 (15)	-0.1027 (16)	0.2459 (9)	0.064 (4)	
C19	1.1206 (14)	0.0448 (15)	0.2018 (10)	0.057 (3)	

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C20	1.0097 (15)	0.1432 (14)	0.2030 (10)	0.059 (3)	
C21	1.2117 (14)	-0.1785 (12)	0.1725 (11)	0.060 (3)	
C22	1.1800 (13)	-0.2823 (11)	0.1474 (10)	0.052 (3)	
C23	1.0266 (11)	-0.3278 (10)	0.1968 (9)	0.043 (2)	
C24	0.7594 (10)	0.2949 (10)	-0.0361 (9)	0.041 (2)	
C25	1.0019 (12)	-0.3678 (9)	0.1130 (10)	0.045 (3)	
C26	0.8641 (12)	0.3473 (10)	-0.0120 (12)	0.053 (3)	
C27	0.8788 (13)	0.3009 (11)	0.0902 (10)	0.049 (3)	
C28	1.0212 (15)	0.2708 (13)	0.1026 (12)	0.061 (3)	
C29	1.1246 (13)	0.3373 (10)	-0.0941 (10)	0.049 (3)	
C30	1.1358 (17)	0.2883 (12)	0.0146 (12)	0.065 (4)	
C31	1.3841 (11)	0.4523 (10)	0.2363 (7)	0.027 (3)	0.82 (2)
C32	1.3342 (11)	0.4954 (11)	0.3232 (7)	0.029 (3)	0.82 (2)
C31A	1.306 (2)	0.430 (3)	0.3017 (15)	0.013 (10)	0.18 (2)
C32A	1.406 (2)	0.515 (3)	0.2605 (15)	0.007 (9)	0.18 (2)
C33	0.7666 (10)	0.1574 (11)	0.4919 (11)	0.030 (5)	0.56 (3)
C34	0.6841 (11)	0.2472 (11)	0.4454 (12)	0.039 (5)	0.56 (3)
C33A	0.7762 (13)	0.2488 (14)	0.4439 (16)	0.039 (7)	0.44 (3)
C34A	0.677 (2)	0.162 (2)	0.4950 (19)	0.034 (6)	0.44 (3)
Cl1	1.5322 (2)	0.5188 (2)	0.12839 (19)	0.0357 (6)	
Cl2	1.2994 (3)	0.3299 (3)	0.2202 (2)	0.0484 (7)	
Cl3	1.4140 (3)	0.6192 (3)	0.3386 (2)	0.0425 (6)	
Cl4	1.1832 (3)	0.4285 (3)	0.4339 (2)	0.0566 (8)	
Cl5	0.9370 (3)	0.2011 (4)	0.4666 (2)	0.0569 (8)	
Cl6	0.7062 (3)	-0.0017 (3)	0.5858 (2)	0.0535 (7)	
Cl7	0.7431 (4)	0.4076 (3)	0.3547 (2)	0.0615 (9)	
Cl8	0.5146 (3)	0.2052 (3)	0.4689 (2)	0.0565 (8)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.053 (7)	0.072 (8)	0.036 (6)	0.006 (6)	-0.008 (5)	-0.043 (6)
C3	0.073 (8)	0.064 (8)	0.012 (4)	-0.005 (6)	0.002 (5)	-0.016 (5)
C6	0.026 (5)	0.042 (6)	0.042 (6)	0.015 (4)	0.011 (4)	-0.021 (5)
C7	0.023 (5)	0.039 (6)	0.065 (7)	0.019 (4)	-0.015 (5)	-0.023 (5)
C8	0.014 (4)	0.046 (6)	0.058 (6)	0.003 (4)	-0.017 (4)	-0.018 (5)
C9	0.007 (4)	0.054 (7)	0.077 (8)	-0.007 (4)	-0.004 (5)	-0.009 (6)
C10	0.026 (6)	0.055 (8)	0.114 (12)	-0.025 (5)	-0.015 (7)	-0.011 (8)
C12	0.040 (6)	0.088 (9)	0.069 (8)	-0.008 (6)	-0.035 (6)	-0.041 (7)
C13	0.039 (6)	0.040 (6)	0.090 (9)	-0.015 (5)	-0.037 (6)	-0.018 (6)
C14	0.038 (6)	0.043 (6)	0.079 (9)	-0.033 (5)	-0.015 (6)	0.024 (6)
C15	0.053 (7)	0.031 (5)	0.035 (6)	-0.016 (5)	0.019 (5)	0.006 (4)
C17	0.121 (12)	0.055 (7)	0.017 (5)	-0.007 (7)	-0.032 (6)	0.001 (5)
C19	0.067 (8)	0.082 (9)	0.054 (7)	0.002 (7)	-0.044 (7)	-0.036 (7)
C20	0.089 (10)	0.066 (8)	0.052 (7)	0.003 (7)	-0.042 (7)	-0.039 (6)
C21	0.072 (8)	0.040 (6)	0.076 (9)	0.003 (6)	-0.055 (7)	0.003 (6)
C22	0.063 (7)	0.041 (6)	0.051 (7)	0.027 (5)	-0.045 (6)	0.000 (5)
C23	0.048 (6)	0.026 (5)	0.042 (6)	-0.001 (4)	-0.020 (5)	0.010 (4)

C24	0.032 (5)	0.024 (5)	0.063 (7)	0.015 (4)	-0.019 (5)	-0.011 (4)
C25	0.053 (6)	0.009 (4)	0.061 (7)	-0.007 (4)	-0.015 (5)	0.004 (4)
C26	0.050 (6)	0.017 (5)	0.090 (9)	0.011 (4)	-0.019 (6)	-0.025 (5)
C27	0.060 (7)	0.035 (6)	0.063 (7)	0.009 (5)	-0.019 (6)	-0.037 (5)
C28	0.080 (9)	0.053 (7)	0.080 (9)	-0.008 (6)	-0.031 (8)	-0.048 (7)
C29	0.065 (8)	0.019 (5)	0.065 (7)	-0.014 (5)	-0.031 (6)	0.003 (5)
C30	0.096 (10)	0.040 (6)	0.079 (9)	-0.036 (7)	-0.034 (8)	-0.020 (6)
C31	0.028 (6)	0.023 (5)	0.028 (6)	-0.005 (4)	-0.009 (5)	-0.001 (4)
C32	0.028 (6)	0.034 (6)	0.027 (6)	0.002 (5)	-0.014 (5)	-0.010 (5)
C31A	0.014 (11)	0.013 (11)	0.014 (11)	0.000 (5)	-0.004 (6)	-0.005 (6)
C32A	0.008 (10)	0.007 (10)	0.006 (11)	0.000 (5)	-0.001 (6)	-0.003 (5)
C33	0.023 (10)	0.039 (11)	0.024 (9)	0.009 (9)	-0.004 (8)	-0.012 (8)
C34	0.051 (13)	0.024 (10)	0.025 (10)	-0.008 (9)	0.008 (9)	-0.006 (8)
C33A	0.042 (15)	0.046 (15)	0.030 (13)	-0.012 (13)	-0.006 (12)	-0.015 (11)
C34A	0.019 (11)	0.028 (12)	0.037 (13)	-0.007 (10)	0.010 (10)	-0.007 (10)
C11	0.0277 (11)	0.0402 (13)	0.0334 (12)	-0.0031 (9)	-0.0002 (9)	-0.0140 (10)
C12	0.0645 (17)	0.0344 (13)	0.0625 (17)	-0.0119 (12)	-0.0345 (14)	-0.0150 (12)
C13	0.0450 (14)	0.0485 (14)	0.0442 (14)	-0.0097 (11)	-0.0119 (11)	-0.0262 (11)
C14	0.0426 (15)	0.074 (2)	0.0378 (14)	-0.0217 (14)	0.0055 (11)	-0.0085 (13)
C15	0.0457 (16)	0.086 (2)	0.0461 (16)	-0.0226 (15)	-0.0169 (13)	-0.0166 (15)
C16	0.0653 (18)	0.0400 (14)	0.0425 (15)	-0.0036 (12)	-0.0213 (13)	0.0076 (11)
C17	0.104 (3)	0.0280 (13)	0.0343 (14)	-0.0175 (14)	-0.0051 (15)	0.0002 (10)
C18	0.0380 (14)	0.0658 (18)	0.0538 (17)	0.0013 (13)	-0.0180 (12)	-0.0021 (14)
C16	0.111 (11)	0.041 (6)	0.022 (5)	-0.027 (7)	-0.020 (6)	0.017 (5)
C5	0.028 (6)	0.092 (10)	0.047 (7)	-0.001 (6)	0.020 (5)	-0.007 (7)
C1	0.046 (6)	0.043 (6)	0.061 (7)	0.017 (5)	-0.009 (5)	-0.041 (6)
C11	0.028 (5)	0.052 (6)	0.054 (6)	-0.004 (4)	-0.033 (5)	-0.001 (5)
C18	0.078 (9)	0.100 (11)	0.028 (6)	0.001 (8)	-0.043 (6)	-0.010 (6)
C4	0.044 (7)	0.100 (11)	0.040 (7)	-0.015 (7)	0.016 (6)	-0.019 (7)

Geometric parameters (Å, °)

C2—C3	1.386 (17)	C23—C25	1.426 (16)
C2—C20	1.426 (17)	C24—C22 ⁱ	1.432 (16)
C2—C1	1.457 (16)	C24—C26	1.433 (16)
C3—C4	1.429 (19)	C25—C29 ⁱ	1.350 (16)
C3—C17	1.482 (19)	C25—C26 ⁱ	1.505 (16)
C6—C1	1.382 (16)	C26—C27	1.348 (17)
C6—C7	1.408 (15)	C26—C25 ⁱ	1.505 (16)
C6—C5	1.504 (18)	C27—C1	1.396 (16)
C7—C24	1.414 (15)	C27—C28	1.468 (17)
C7—C8	1.454 (14)	C28—C30	1.299 (19)
C8—C11	1.394 (15)	C29—C25 ⁱ	1.350 (16)
C8—C9	1.427 (15)	C29—C30	1.415 (17)
C9—C10	1.347 (17)	C29—C14 ⁱ	1.463 (19)
C9—C5	1.55 (2)	C31—C32	1.280 (2)
C10—C13 ⁱ	1.364 (19)	C31—C11	1.699 (10)
C10—C14	1.487 (18)	C31—C12	1.735 (10)

supplementary materials

C12—C13	1.379 (18)	C32—C13	1.709 (10)
C12—C19	1.443 (17)	C32—C14	1.735 (10)
C12—C11 ⁱ	1.471 (16)	C31A—C32A	1.282 (2)
C13—C10 ⁱ	1.364 (19)	C31A—C12	1.747 (9)
C13—C30	1.529 (19)	C31A—C14	1.748 (9)
C14—C15	1.354 (19)	C32A—C13	1.751 (9)
C14—C29 ⁱ	1.463 (19)	C32A—C11	1.751 (9)
C15—C16	1.480 (19)	C33—C34	1.281 (2)
C15—C4	1.517 (19)	C33—C15	1.728 (8)
C17—C18	1.372 (19)	C33—C16	1.731 (8)
C17—C16	1.427 (17)	C34—C18	1.720 (8)
C19—C20	1.360 (18)	C34—C17	1.726 (8)
C19—C18	1.454 (19)	C33A—C34A	1.281 (2)
C20—C28	1.510 (19)	C33A—C15	1.711 (9)
C21—C22	1.327 (17)	C33A—C17	1.721 (9)
C21—C11 ⁱ	1.426 (16)	C34A—C18	1.75 (2)
C21—C18	1.524 (19)	C34A—C16	1.75 (2)
C22—C24 ⁱ	1.432 (16)	C5—C4	1.251 (19)
C22—C23	1.525 (16)	C11—C21 ⁱ	1.426 (16)
C23—C16	1.358 (17)	C11—C12 ⁱ	1.471 (16)
C3—C2—C20	120.2 (11)	C27—C26—C24	121.6 (11)
C3—C2—C1	119.0 (11)	C27—C26—C25 ⁱ	117.9 (11)
C20—C2—C1	108.6 (11)	C24—C26—C25 ⁱ	108.2 (10)
C2—C3—C4	120.7 (12)	C26—C27—C1	118.9 (11)
C2—C3—C17	119.2 (12)	C26—C27—C28	121.0 (11)
C4—C3—C17	107.2 (11)	C1—C27—C28	108.6 (11)
C1—C6—C7	119.7 (10)	C30—C28—C27	120.3 (13)
C1—C6—C5	121.1 (11)	C30—C28—C20	121.0 (13)
C7—C6—C5	107.4 (11)	C27—C28—C20	106.3 (11)
C6—C7—C24	118.8 (10)	C25 ⁱ —C29—C30	123.2 (12)
C6—C7—C8	111.1 (9)	C25 ⁱ —C29—C14 ⁱ	117.5 (11)
C24—C7—C8	117.6 (10)	C30—C29—C14 ⁱ	108.4 (11)
C11—C8—C9	119.7 (10)	C28—C30—C29	120.3 (14)
C11—C8—C7	118.9 (9)	C28—C30—C13	119.0 (12)
C9—C8—C7	109.3 (10)	C29—C30—C13	107.8 (12)
C10—C9—C8	121.5 (12)	C32—C31—C11	121.3 (8)
C10—C9—C5	120.3 (12)	C32—C31—C12	121.7 (8)
C8—C9—C5	106.4 (10)	C11—C31—C12	117.0 (5)
C9—C10—C13 ⁱ	119.8 (13)	C31—C32—C13	122.2 (9)
C9—C10—C14	117.8 (14)	C31—C32—C14	121.7 (9)
C13 ⁱ —C10—C14	110.6 (12)	C13—C32—C14	116.0 (5)
C13—C12—C19	119.7 (12)	C32A—C31A—C12	117.9 (7)
C13—C12—C11 ⁱ	118.3 (11)	C32A—C31A—C14	116.8 (7)
C19—C12—C11 ⁱ	109.1 (11)	C12—C31A—C14	125.3 (7)
C10 ⁱ —C13—C12	123.0 (12)	C31A—C32A—C13	119.1 (7)

C10 ⁱ —C13—C30	107.0 (11)	C31A—C32A—C11	118.2 (7)
C12—C13—C30	119.8 (12)	C13—C32A—C11	122.7 (6)
C15—C14—C29 ⁱ	121.7 (11)	C34—C33—C15	119.1 (7)
C15—C14—C10	119.8 (13)	C34—C33—C16	121.0 (7)
C29 ⁱ —C14—C10	106.2 (12)	C15—C33—C16	119.8 (5)
C14—C15—C16	119.4 (11)	C33—C34—C18	119.7 (7)
C14—C15—C4	121.6 (12)	C33—C34—C17	120.9 (7)
C16—C15—C4	105.7 (11)	C18—C34—C17	119.3 (5)
C18—C17—C16	119.9 (14)	C34A—C33A—C15	118.4 (14)
C18—C17—C3	119.0 (12)	C34A—C33A—C17	119.0 (14)
C16—C17—C3	109.9 (13)	C15—C33A—C17	122.6 (7)
C20—C19—C12	120.6 (13)	C33A—C34A—C18	119.4 (17)
C20—C19—C18	119.7 (12)	C33A—C34A—C16	120.8 (17)
C12—C19—C18	108.0 (11)	C18—C34A—C16	119.8 (7)
C19—C20—C2	121.4 (12)	C23—C16—C17	121.5 (13)
C19—C20—C28	119.8 (13)	C23—C16—C15	117.9 (11)
C2—C20—C28	106.7 (11)	C17—C16—C15	108.4 (12)
C22—C21—C11 ⁱ	120.2 (13)	C4—C5—C6	118.8 (15)
C22—C21—C18	119.0 (12)	C4—C5—C9	122.0 (14)
C11 ⁱ —C21—C18	107.1 (11)	C6—C5—C9	105.8 (11)
C21—C22—C24 ⁱ	121.0 (12)	C6—C1—C27	122.1 (11)
C21—C22—C23	119.6 (11)	C6—C1—C2	117.2 (10)
C24 ⁱ —C22—C23	108.4 (10)	C27—C1—C2	109.7 (10)
C16—C23—C25	121.6 (11)	C8—C11—C21 ⁱ	121.5 (11)
C16—C23—C22	119.6 (11)	C8—C11—C12 ⁱ	117.6 (10)
C25—C23—C22	106.5 (9)	C21 ⁱ —C11—C12 ⁱ	108.6 (11)
C7—C24—C22 ⁱ	120.8 (10)	C17—C18—C19	120.6 (13)
C7—C24—C26	118.8 (10)	C17—C18—C21	120.2 (13)
C22 ⁱ —C24—C26	108.7 (10)	C19—C18—C21	107.1 (11)
C29 ⁱ —C25—C23	121.9 (11)	C5—C4—C3	123.1 (15)
C29 ⁱ —C25—C26 ⁱ	117.2 (11)	C5—C4—C15	118.2 (15)
C23—C25—C26 ⁱ	108.2 (9)	C3—C4—C15	108.9 (12)
C20—C2—C3—C4	-137.2 (11)	C15—C33—C34—C18	179.6 (9)
C1—C2—C3—C4	0.8 (15)	C16—C33—C34—C18	-4(2)
C20—C2—C3—C17	-0.7 (14)	C15—C33—C34—C17	2(2)
C1—C2—C3—C17	137.3 (10)	C16—C33—C34—C17	178.3 (10)
C1—C6—C7—C24	0.7 (13)	C15—C33A—C34A—C18	-177.4 (12)
C5—C6—C7—C24	-142.6 (9)	C17—C33A—C34A—C18	2(3)
C1—C6—C7—C8	141.9 (9)	C15—C33A—C34A—C16	0(3)
C5—C6—C7—C8	-1.4 (10)	C17—C33A—C34A—C16	179.4 (12)
C6—C7—C8—C11	-142.2 (9)	C32—C31—C11—C32A	0(2)
C24—C7—C8—C11	-0.4 (13)	C12—C31—C11—C32A	177 (3)
C6—C7—C8—C9	0.3 (10)	C31A—C32A—C11—C31	0(2)
C24—C7—C8—C9	142.0 (9)	C13—C32A—C11—C31	179 (4)
C11—C8—C9—C10	0.1 (14)	C32—C31—C12—C31A	-2(3)
C7—C8—C9—C10	-141.9 (10)	C11—C31—C12—C31A	-179 (3)

supplementary materials

C11—C8—C9—C5	143.0 (9)	C32A—C31A—C12—C31	2(2)
C7—C8—C9—C5	1.0 (10)	C14—C31A—C12—C31	180 (5)
C8—C9—C10—C13 ⁱ	0.0 (15)	C31—C32—C13—C32A	1(3)
C5—C9—C10—C13 ⁱ	-137.9 (11)	C14—C32—C13—C32A	180 (3)
C8—C9—C10—C14	139.6 (11)	C31A—C32A—C13—C32	-1(2)
C5—C9—C10—C14	1.7 (15)	C11—C32A—C13—C32	-180 (4)
C19—C12—C13—C10 ⁱ	-138.2 (11)	C31—C32—C14—C31A	0(2)
C11 ⁱ —C12—C13—C10 ⁱ	-0.8 (15)	C13—C32—C14—C31A	-179 (3)
C19—C12—C13—C30	2.4 (15)	C32A—C31A—C14—C32	0(2)
C11 ⁱ —C12—C13—C30	139.8 (10)	C12—C31A—C14—C32	-178 (5)
C9—C10—C14—C15	-1.0 (15)	C34A—C33A—C15—C33	2.6 (16)
C13 ⁱ —C10—C14—C15	142.1 (11)	C17—C33A—C15—C33	-177 (3)
C9—C10—C14—C29 ⁱ	-143.9 (10)	C34—C33—C15—C33A	0.4 (18)
C13 ⁱ —C10—C14—C29 ⁱ	-0.8 (12)	C16—C33—C15—C33A	-176 (2)
C29 ⁱ —C14—C15—C16	-0.6 (15)	C34—C33—C16—C34A	-0.7 (18)
C10—C14—C15—C16	-137.7 (11)	C15—C33—C16—C34A	175 (3)
C29 ⁱ —C14—C15—C4	134.6 (11)	C33A—C34A—C16—C33	-2.6 (17)
C10—C14—C15—C4	-2.5 (16)	C18—C34A—C16—C33	174 (3)
C2—C3—C17—C18	1.1 (15)	C34A—C33A—C17—C34	-1.0 (17)
C4—C3—C17—C18	142.8 (10)	C15—C33A—C17—C34	179 (3)
C2—C3—C17—C16	-142.6 (10)	C33—C34—C17—C33A	-2.1 (18)
C4—C3—C17—C16	-0.9 (12)	C18—C34—C17—C33A	-179 (3)
C13—C12—C19—C20	-1.9 (15)	C33—C34—C18—C34A	3.8 (17)
C11 ⁱ —C12—C19—C20	-142.7 (10)	C17—C34—C18—C34A	-179 (3)
C13—C12—C19—C18	141.0 (10)	C33A—C34A—C18—C34	-0.7 (18)
C11 ⁱ —C12—C19—C18	0.2 (11)	C16—C34A—C18—C34	-178 (3)
C12—C19—C20—C2	138.1 (11)	C25—C23—C16—C17	-137.0 (11)
C18—C19—C20—C2	-0.6 (16)	C22—C23—C16—C17	0.0 (15)
C12—C19—C20—C28	0.6 (15)	C25—C23—C16—C15	1.1 (14)
C18—C19—C20—C28	-138.1 (11)	C22—C23—C16—C15	138.1 (10)
C3—C2—C20—C19	0.5 (15)	C18—C17—C16—C23	-1.6 (16)
C1—C2—C20—C19	-141.3 (10)	C3—C17—C16—C23	141.8 (11)
C3—C2—C20—C28	142.7 (10)	C18—C17—C16—C15	-143.1 (11)
C1—C2—C20—C28	0.9 (11)	C3—C17—C16—C15	0.2 (12)
C11 ⁱ —C21—C22—C24 ⁱ	1.4 (16)	C14—C15—C16—C23	-1.2 (14)
C18—C21—C22—C24 ⁱ	136.9 (11)	C4—C15—C16—C23	-142.6 (10)
C11 ⁱ —C21—C22—C23	-138.4 (11)	C14—C15—C16—C17	142.0 (10)
C18—C21—C22—C23	-2.8 (15)	C4—C15—C16—C17	0.5 (11)
C21—C22—C23—C16	2.3 (14)	C1—C6—C5—C4	1.0 (16)
C24 ⁱ —C22—C23—C16	-142.0 (10)	C7—C6—C5—C4	143.6 (11)
C21—C22—C23—C25	145.0 (10)	C1—C6—C5—C9	-140.7 (10)
C24 ⁱ —C22—C23—C25	0.7 (10)	C7—C6—C5—C9	1.9 (10)
C6—C7—C24—C22 ⁱ	137.9 (10)	C10—C9—C5—C4	1.5 (17)
C8—C7—C24—C22 ⁱ	-0.8 (13)	C8—C9—C5—C4	-141.9 (12)
C6—C7—C24—C26	-1.1 (13)	C10—C9—C5—C6	141.7 (10)

C8—C7—C24—C26	-139.9 (9)	C8—C9—C5—C6	-1.8 (10)
C16—C23—C25—C29 ⁱ	0.8 (15)	C7—C6—C1—C27	-0.6 (14)
C22—C23—C25—C29 ⁱ	-141.0 (9)	C5—C6—C1—C27	137.6 (10)
C16—C23—C25—C26 ⁱ	141.4 (10)	C7—C6—C1—C2	-141.0 (9)
C22—C23—C25—C26 ⁱ	-0.4 (10)	C5—C6—C1—C2	-2.8 (14)
C7—C24—C26—C27	1.6 (14)	C26—C27—C1—C6	1.1 (15)
C22 ⁱ —C24—C26—C27	-141.9 (10)	C28—C27—C1—C6	-142.7 (9)
C7—C24—C26—C25 ⁱ	143.1 (9)	C26—C27—C1—C2	144.0 (10)
C22 ⁱ —C24—C26—C25 ⁱ	-0.4 (11)	C28—C27—C1—C2	0.2 (11)
C24—C26—C27—C1	-1.6 (15)	C3—C2—C1—C6	1.9 (14)
C25 ⁱ —C26—C27—C1	-139.5 (10)	C20—C2—C1—C6	144.2 (9)
C24—C26—C27—C28	137.6 (11)	C3—C2—C1—C27	-143.1 (9)
C25 ⁱ —C26—C27—C28	-0.3 (14)	C20—C2—C1—C27	-0.7 (11)
C26—C27—C28—C30	-0.1 (16)	C9—C8—C11—C21 ⁱ	-137.7 (10)
C1—C27—C28—C30	142.9 (11)	C7—C8—C11—C21 ⁱ	0.8 (14)
C26—C27—C28—C20	-142.6 (10)	C9—C8—C11—C12 ⁱ	0.2 (12)
C1—C27—C28—C20	0.3 (11)	C7—C8—C11—C12 ⁱ	138.7 (9)
C19—C20—C28—C30	0.0 (16)	C16—C17—C18—C19	138.8 (11)
C2—C20—C28—C30	-142.9 (11)	C3—C17—C18—C19	-1.2 (15)
C19—C20—C28—C27	142.2 (10)	C16—C17—C18—C21	1.0 (16)
C2—C20—C28—C27	-0.8 (11)	C3—C17—C18—C21	-139.1 (11)
C27—C28—C30—C29	0.3 (16)	C20—C19—C18—C17	1.0 (16)
C20—C28—C30—C29	137.3 (12)	C12—C19—C18—C17	-142.3 (10)
C27—C28—C30—C13	-136.5 (11)	C20—C19—C18—C21	143.7 (10)
C20—C28—C30—C13	0.6 (16)	C12—C19—C18—C21	0.4 (11)
C25 ⁱ —C29—C30—C28	-0.2 (17)	C22—C21—C18—C17	1.3 (16)
C14 ⁱ —C29—C30—C28	-143.2 (11)	C11 ⁱ —C21—C18—C17	142.0 (11)
C25 ⁱ —C29—C30—C13	140.8 (10)	C22—C21—C18—C19	-141.5 (10)
C14 ⁱ —C29—C30—C13	-2.2 (11)	C11 ⁱ —C21—C18—C19	-0.8 (11)
C10 ⁱ —C13—C30—C28	144.3 (11)	C6—C5—C4—C3	1.9 (18)
C12—C13—C30—C28	-1.8 (15)	C9—C5—C4—C3	137.2 (13)
C10 ⁱ —C13—C30—C29	2.7 (11)	C6—C5—C4—C15	-140.2 (11)
C12—C13—C30—C29	-143.5 (10)	C9—C5—C4—C15	-4.9 (17)
C11—C31—C32—C13	-1.3 (14)	C2—C3—C4—C5	-2.8 (18)
C12—C31—C32—C13	-178.3 (6)	C17—C3—C4—C5	-143.9 (12)
C11—C31—C32—C14	180.0 (6)	C2—C3—C4—C15	142.2 (10)
C12—C31—C32—C14	3.0 (14)	C17—C3—C4—C15	1.2 (12)
C12—C31A—C32A—C13	178.6 (19)	C14—C15—C4—C5	5.5 (16)
C14—C31A—C32A—C13	1(4)	C16—C15—C4—C5	145.9 (11)
C12—C31A—C32A—C11	-2(4)	C14—C15—C4—C3	-141.5 (11)
C14—C31A—C32A—C11	179.8 (19)	C16—C15—C4—C3	-1.1 (12)

Symmetry codes: (i) $-x+2, -y, -z$.

Fig. 1

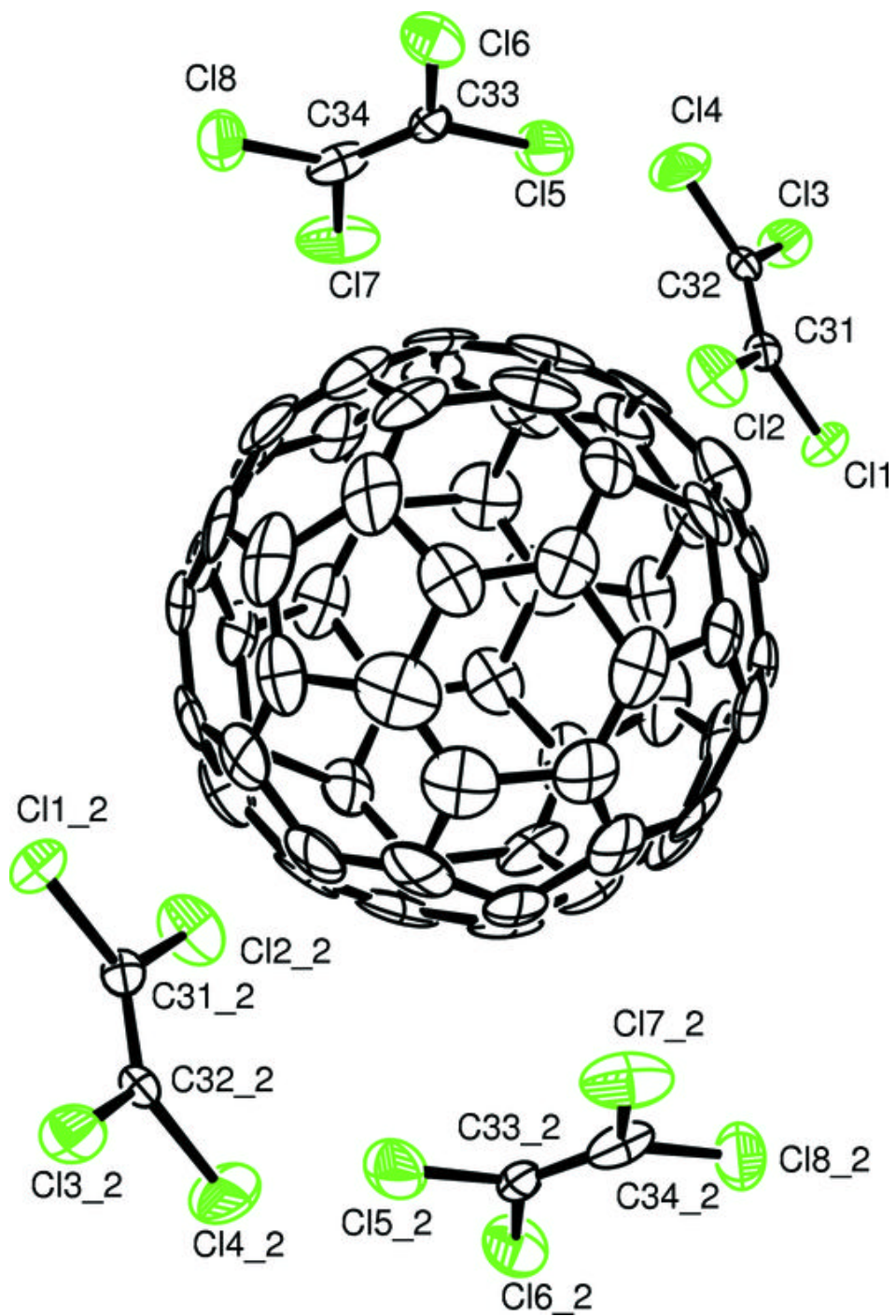


Fig. 2

