Supplementary Material (ESI) for Chemical Communications

Supporting Information for:

Synthesis and Characterization of 2,7-bis(pentafluorophenylethynyl)-hexafluoroheterofluorenes: New Materials with High Electron Affinities

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1) **Experimental**

General. All reactions were performed under an inert atmosphere of nitrogen using standard Schlenk techniques. Tetrahydrofuran (THF) was dried over Na and stored under nitrogen. Hexanes, Et_2O and toluene were dried on a VAC systems solvent purifier column and stored under nitrogen. Dichlorophenylphosphine, dichlorothethylphosphine, hydrogen peroxide (30% in water), butyl lithium (1.6 M in hexanes), tetrabutylammonium hexafluorophosphate (Aldrich), dibromotetrafluorobenzene (Oakwood Products Inc) and dichlorodiphenylgermane, dichlorodiethylgermane (Gelest) were used as received. Dichlorodiphenylsilane and dichlorodimethylsilane (Gelest) was distilled prior to use. UV-vis spectra were measured with a Varian Cary 300 Bio UV-vis spectrometer and all samples were measured in THF unless otherwise stated. Emission spectra were measured with the Varian Cary Eclipse Spectrometer. Fluorescence quantum yields (Φ_{PL}) were calculated with respect to freshly sublimed 9,9-diphenylanthracene in THF (Φ = 0.90). Unless otherwise stated, ^{1}H, ^{31}P and ^{19}F NMR spectra were measured in $CDCl_3$ with a Bruker AVQ 400 MHz spectrometer. All chemical shifts are reported in ppm units. For ^{19}F NMR spectra C_6F_6 was used as internal reference at -163 ppm and ^{1}H NMR chemical shifts were referenced to the residual peak of the deuterated solvent at 7.26 ppm for $CDCl_3$. Melting point determinations were performed by a Melt Temp II instrument and are uncorrected. Cyclic voltammetry was performed on a Bioanalytical Systems CV-50W Voltammetric Analyzer with a C-3 Cell Stand. The potentials were measured vs a $Ag/AgNO_3$ nonaqueous reference electrode, with Pt disc (PTE) as working electrode and a Pt wire axial electrode, in acetonitrile containing 0.1 mol/L tetrabutylammoniumhexafluorophosphate, with ferrocene as external standard (HOMO =

-4.8 eV) and potential sweep rate of 100 mV/s unless otherwise stated. All elemental analyses were obtained at the Micro-Mass Facility of the University of California, Berkeley. Lithium triisopropylsilylacetylene was made from triisopropylsilylacetylene and BuLi in hexanes and 2,2′-dibromooctafluorobiphenyl was prepared as described in the literature.[1]

2,2′-dibromo-4,4′-bis(pentafluorophenylethynyl)-hexafluorobiphenyl (1). To a solution of 2,2′-dibromooctafluorobiphenyl (0.230 g, 0.5 mmol) in toluene (25 mL) at room temperature a solution of lithium triisopropylsilylacetylene (0.210 g, 1.1 mmol) in THF (8 mL) was added. The resulting solution was stirred for 3 - 5 days. The resulting yellow solution was washed with water (2 x 30 mL). The organic components were dried over $MgSO_4$ and filtered through celite. The resulting solution was then diluted with wet toluene (600 mL) and distilled THF (45 mL). The solution was sparged with nitrogen for 20 min and then Bu_4NF (1.1 mL, 1.0 M in THF 1% H_2O) was added, and then the resulting solution was stirred for 5 minutes. The light brown solution was washed with water (2 x 150 mL) and the organic components were dried over $MgSO_4$ and passed through a silica plug. The resulting solution was concentrated via rotary evaporation. *Note: It is critical at this step to not mix concentrated solutions of unprotected diyne with THF or CH_2Cl_2. This will result in decomposition of the compound.* To the concentrated solution was added NEt_3 (25 mL) and iodopentafluorobenzene (0.730 g, 2.5 mmol) and the solution was sparged with nitrogen for 10 minutes. The degassed solution was added to a Schlenk flask containing $PdCl_2(PPh_3)_2$ (0.020 g, 0.03 mmol), CuI (0.020 g, 0.1 mmol) and PPh_3 (0.015 g, 0.05 mmol) and the entire mixture was heated at 40 °C for 12 h, with vigorous stirring, under a flow of nitrogen. Toluene (20 mL) was added to the

solution and the mixture was washed with aqueous NH_4Cl (2 x 20 mL) and water (2 x 20 mL). The collected organic components were dried with $MgSO_4$, filtered through celite and concentrated via rotary evaporation. The disubstituted product was isolated via column chromatography (hexanes) to give 0.190 g (0.24 mmol, 47 %) of **1** as a white powder. ^{19}F NMR ($CDCl_3$, 400 MHz, 25 °C) δ -101.74 (1F, m), -129.48 (1F, m), -135.44 (2F, m), -136.82 (1F, m), -150.37 (1F, m), -161.88 (2F, m). Anal. Calcd for $C_{28}Br_2F_{16}$: C, 42.03. Found: C, 42.05. mp > 250 °C dec

2,7-bis(pentafluorophenylethynyl)-9,9-diphenyl-hexafluorosilafluorene (2a). 2,2′-dibromo-4,4′-bis(pentafluorophenylethynyl)hexafluorobiphenyl (0.055 g, 0.07 mmol) was dissolved in toluene/Et_2O (10 mL/ 2 mL) and the mixture was cooled to – 78 °C. To this was added butyllithium (0.08 mL, 1.6 M in hexanes) and the resulting solution was stirred for 20 min at -78 °C, after which dichlorodiphenylsilane (0.05 mL) in THF (1 mL) was added and the entire solution was allowed to warm to room temperature over 12 h with stirring. The resulting yellow solution was quenched with water (2 x 15 mL) and the organic components were extracted into CH_2Cl_2 (2 x 20 mL). The combined extracts were dried over $MgSO_4$, filtered through celite and concentrated via rotary evaporation. The disubstituted product was isolated via recrystallization from hot hexanes to give trace amounts of **2a** as a white powder. ^1H NMR ($CDCl_3$, 400 MHz, 25 °C) δ 7.455 (2H, m), 7.529 (1H, m), 7.67 (2H, m); ^{19}F NMR ($CDCl_3$, 400 MHz, 25 °C) δ -98.21 (1F, m), -123.15 (1F, m), -131.81 (1F, m), -135.76 (2F, m), -151.09 (1F, m), -162.16 (2F, m). Insufficient amounts of pure **2a** were isolated to allow full characterization thus identity and purity are shown by ^1H and ^{19}F NMR.

2,7-bis(pentafluorophenylethynyl)-9,9-dimethyl-hexafluorosilafluorene (2b). 2,2′-dibromo-4,4′-bis(pentafluorophenylethynyl)-hexafluorobiphenyl (0.050 g, 0.06 mmol) was dissolved in toluene/Et_2O (10 mL/ 2 mL) and the mixture was cooled to – 78 °C. To this was added butyllithium (0.08 mL, 1.6 M in hexanes) and the resulting solution was stirred for 20 min at -78 °C, after which dichlorodimethylsilane (0.05 mL) was added and the entire solution was allowed to warm to room temperature over 12 h with stirring. The resulting yellow solution was quenched with water (2 x 15 mL) and the organic components were extracted into CH_2Cl_2 (2 x 20 mL). The extracts were dried over $MgSO_4$, filtered through celite and concentrated via rotary evaporation. The disubstituted product was isolated via recrystallization from hot hexanes to give 0.026 g (0.04 mmol, 60 %) of **2b** as a white powder. ^{1}H NMR ($CDCl_3$, 400 MHz, 25 °C) δ 0.627 (s); ^{19}F NMR ($CDCl_3$, 400 MHz, 25 °C) δ -102.35 (1F, m), -124.41 (1F, m), -132.68 (1F, m), -135.91 (2F, m), -151.23 (1F, m), -162.21 (2F, m). Anal. Calcd for $C_{30}H_6SiF_{16}$: C, 51.59; H, 0.87. Found: C, 51.36; H, 0.54. m.p. 227-229 °C

2,7-bis(pentafluorophenylethynyl)-9,9-diphenyl-hexafluorogermafluorene (3a). 2,2′-dibromo-4,4′-bis(pentafluorophenylethynyl)hexafluorobiphenyl (0.075 g, 0.09 mmol) was dissolved in toluene/Et_2O (10 mL/ 2 mL) and the mixture was cooled to – 78 °C. To this was added butyllithium (0.12 mL, 1.6 M in hexanes) and the resulting solution was stirred for 20 min at -78 °C, after which dichlorodiphenylgermane (0.1 mL) in THF (1 mL) was added and the entire solution was allowed to warm to room temperature over 12 h with stirring. The resulting yellow solution was quenched with water (2 x 15 mL) and the organic components were extracted into CH_2Cl_2 (2 x 20 mL). The extracts were dried over $MgSO_4$, filtered through celite and concentrated via rotary

evaporation. The disubstituted product was isolated via recrystallization from hot hexanes to give 0.046 g (0.05 mmol, 57 %) of **3a** as a white powder. ^{1}H NMR ($CDCl_3$, 400 MHz, 25 °C) δ 7.422 – 7.492 (3H, m), 7.603 (2H, m); ^{19}F NMR ($CDCl_3$, 400 MHz, 25 °C) δ -98.50 (1F, m), -124.44 (1F, m), -130.92 (1F, m), -135.76 (2F, m), -151.09 (1F, m), -162.17 (2F, m). Anal. Calcd for $C_{40}H_{10}GeF_{16}$: C, 55.41; H, 1.16. Found: C, 55.76; H, 0.96. m.p. 277- 280 °C

2,7-bis(pentafluorophenylethynyl)-9,9-diethyl-hexafluorogermafluorene (3b). 2,2′-dibromo-4,4′-bis(pentafluorophenylethynyl)hexafluorobiphenyl (75 mg, 0.09 mmol) was dissolved in toluene/Et_2O (10 mL/ 2 mL) and cooled to – 78 °C. To this was added butyllithium (0.12 mL, 1.6 M in hexanes) and the solution was stirred for 20 min at -78 °C, after which dichlorodiethylgermane (0.1 mL) in THF (1.5 mL) was added and the entire solution was allowed to warm to room temperature over 12 hours. The resulting yellow solution was quenched with water (2 x 15 mL) and the organic components were extracted into CH_2Cl_2. The extracts were dried over $MgSO_4$ and concentrated via rotary evaporation. The disubstituted product was isolated via recrystallization from hot hexanes to give 56 mg (60 %, 0.06 mmol) of **3b** as a white powder. ^{1}H NMR ($CDCl_3$, 400 MHz, 25 °C) δ 1.107 (3H, t), 1.439 (2H, q); ^{19}F NMR ($CDCl_3$, 400 MHz, 25 °C) δ -99.80 (1F, m), -125.81 (1F, m), -131.73 (1F, m), -135.92 (2F, m), -151.32 (1F, m), -162.28 (2F, m). Anal. Calcd for $C_{32}H_{10}GeF_{16}$: C, 49.85; H, 1.31. Found: C, 49.94; H, 1.22. m.p. 237-240 °C

2,7-bis(pentafluorophenylethynyl)-9-phenyl-hexafluorophosphafluorene oxide (4a). 2,2′-dibromo-4,4′-bis(pentafluorophenylethynyl)hexafluorobiphenyl (0.060 g, 0.07 mmol) was dissolved in toluene/Et_2O (10 mL/ 2 mL) and the mixture was cooled to – 78

°C. To this was added butyllithium (0.08 mL, 1.6 M in hexanes) and the resulting solution was stirred for 20 min at -78 °C, after which dichloroethylphosphine (0.05 mL) was added and the entire solution was allowed to warm to room temperature over 12 h with stirring. The resulting yellow solution was quenched with water (2 x 15 mL) and the organic components were extracted into CH_2Cl_2 (2 x 20 mL). H_2O_2 (0.1 mL, 30 wt% solution in H_2O) was added and the mixture was stirred at room temperature for 4 h. The light yellow solution was washed with water (2 x 25 mL) and the organic components were dried with $MgSO_4$, filtered through celite and dried under rotary evaporation. The disubstituted product was isolated via slow evaporation of CH_2Cl_2 from hot hexanes/CH_2Cl_2 to give 0.030 g (0.04 mmol, 55 %) of **4a** as a white powder. ^{1}H NMR ($CDCl_3$, 400 MHz, 25 °C) δ 7.555 (2H, td), 7.672 (1H,t), 7.765 (2H, dd); ^{19}F NMR ($CDCl_3$, 400 MHz, 25 °C) δ -104.68 (1F, m), -118.06 (1F, m), -131.55 (1F, m), -135.29 (2F, m), -149.95 (1F, m), -161.75 (2F, m); ^{31}P ($CDCl_3$, 161.6 MHz, 25 °C) δ 26.32. Anal. Calcd for $C_{34}H_5POF_{16}$: C, 53.43; H, 0.66. Found: C, 53.32; H, 0.57. m.p. 301- 303 °C

2,7-bis(pentafluorophenylethynyl)-9-ethyl-hexafluorophosphafluorene oxide (4b) 2,2'-dibromo-4,4'-bis(pentafluorophenylethynyl)hexafluorobiphenyl (0.060 g, 0.07 mmol) was dissolved in toluene/Et_2O (10 mL/ 2 mL) and the mixture was cooled to – 78 °C. To this was added butyllithium (0.08 mL, 1.6 M in hexanes) and the solution was stirred for 20 min at -78 °C, after which dichloroethylphophine (0.05 mL) was added and the entire solution was allowed to warm to room temperature over 12 h with stirring. The resulting yellow solution was quenched with water (2 x 15 mL) and the organic components were extracted into CH_2Cl_2 (2 x 20 mL). H_2O_2 (0.1 mL, 30 wt% solution in H_2O) was added and the mixture was stirred at room temperature for 4 h. The light

yellow solution was washed with water (2 x 25 mL) and the organic components were dried with $MgSO_4$, filtered through celite and dried under rotary evaporation. The disubstituted product was isolated via slow evaporation of CH_2Cl_2 from hot hexanes/CH_2Cl_2 to give 0.030 g (0.04 mmol, 55 %) of **4b** as a white powder. ^{1}H NMR ($CDCl_3$, 400 MHz, 25 °C) δ 1.175 (3H, dt), 2.495 (2H,m); ^{19}F NMR ($CDCl_3$, 400 MHz, 25 °C) δ -105.45 (1F, m), -118.33 (1F, m), -131.42 (1F, m), -135.25 (2F, m), -149.90 (1F, m), -161.71 (2F, m), ^{31}P ($CDCl_3$, 161.6 MHz, 25 °C) δ 41.62. Anal. Calcd for $C_{30}H_5POF_{16}$: C, 50.30; H, 0.70. Found: C, 50.36; H, 0.61. m.p. 266 – 267 °C

Solar Cell Device Construction and Measurement

150 nm sputtered ITO-coated (20 Ω m^{-1}) glass substrates were obtained from Thin Film Devices, Inc. The substrates were subjected to ultrasonication for 20 min in acetone, and then 2% Helmanex soap in water for 20 min, followed by extensive rinsing with deionized water and ultrasonication in deionized water and then 2-propanol. The substrates were then dried under a stream of nitrogen before treatment for 10 min in an oxygen plasma at 300 mtorr (1 torr = 133.28 Pa) at high power. PEDOT:PSS in water (Baytron-P) was filtered through a 0.45 μm filter and spin-coated onto the ITO surface at 4000 rpm for 60 s and baked for 1 h at 125 °C, affording a 30 nm layer.. All procedures after this point were performed in an inert-atmosphere glove box. Solutions of P3HT, **3b** and **4b** were prepared separately in $CHCl_3$ (10 mg mL^{-1}). Immediately prior to deposition, the solutions were passed though a 0.2 μm polytetrafluoroethylene syringe filter. For a 1:1 P3HT/**3b** or P3HT/**4b** device, equal amounts of each solution were combined. The blend solution was applied to the substrate and spun at 1000 rpm for 60 s. A small portion of the organic layer was removed with tweezers to allow contact with the

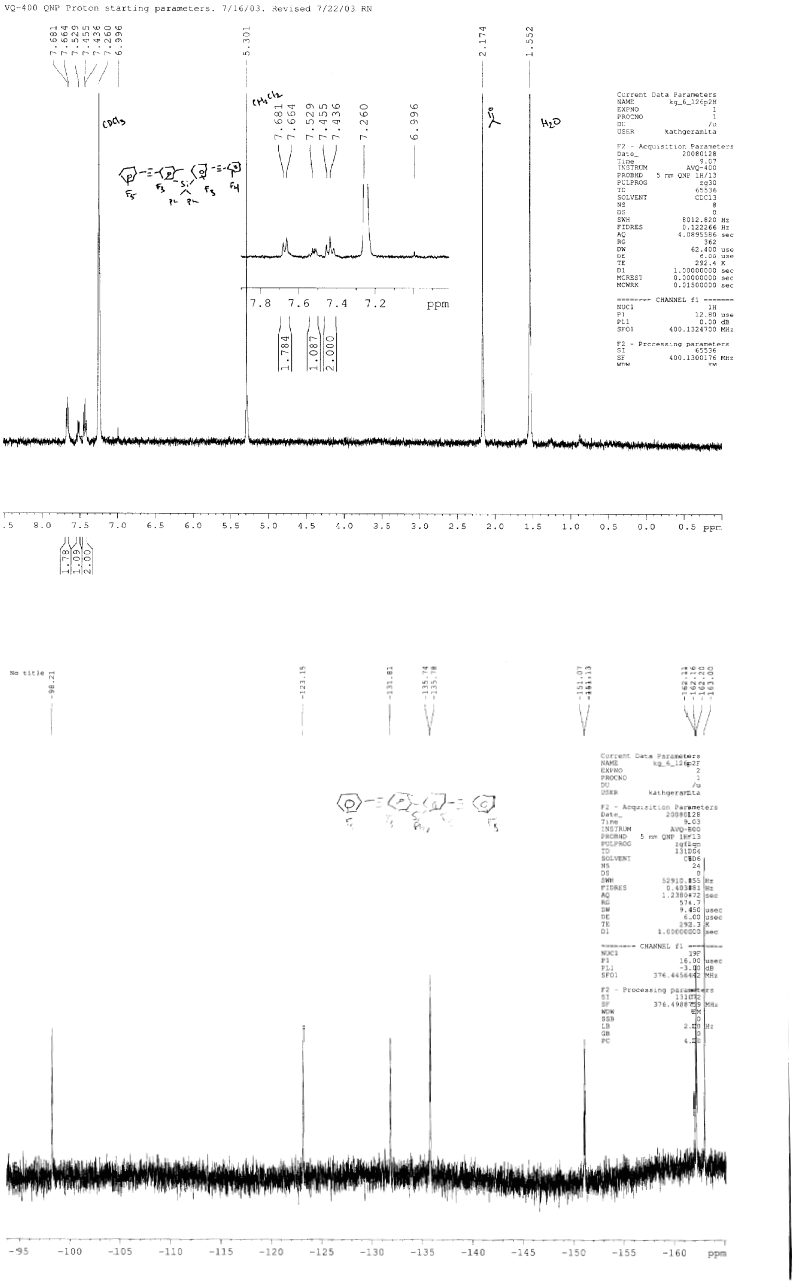
ITO. The substrates were then placed in a resistive-heating evaporation chamber and held under vacuum (10^{-7} torr) for 4 h before evaporating 100 nm of Al through a shadow mask at a rate of 0.1–0.5 nms^{-1} while rotating the substrates at approximately 1 Hz to ensure even electrode deposition. The configuration of the shadow mask afforded eight independent devices on each substrate and one rectangular pad to connect the Al to the ITO substrate. Devices were left to cool to room temperature before further processing. Testing of the devices was performed under an argon atmosphere with an oriel xenon arc lamp with an AM 1.5G solar filter. Current–voltage behavior was measured with a Keithly 236 SMU instrument. Reported efficiencies are the average from 8 independent measurements made on one spin coated device.

References:

1. Cohen, S.; Fenton, D.; Tomlinson, A.; Massey, A. *J. Organometal. Chem.* **1966**, *6*, 301.

2) ^{1}H and ^{19}F NMR Spectra of Compounds 2a

Compound 2a



3) Optical Spectroscopy Data

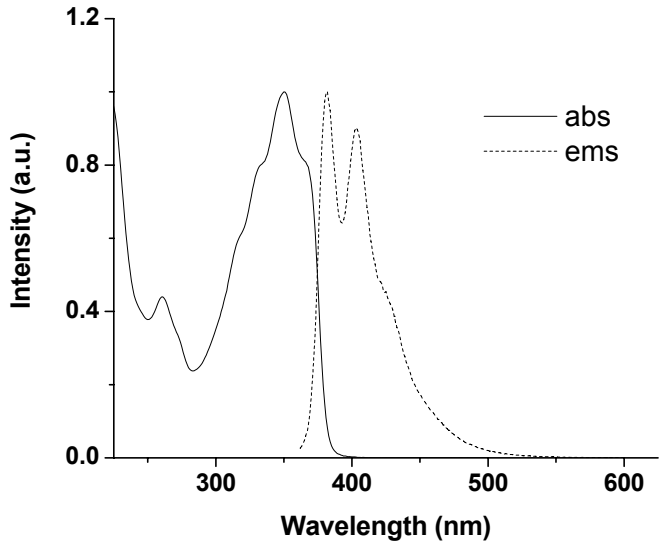


Figure S.1 Normalized Absorption and Emission Spectra for **2a** in THF.

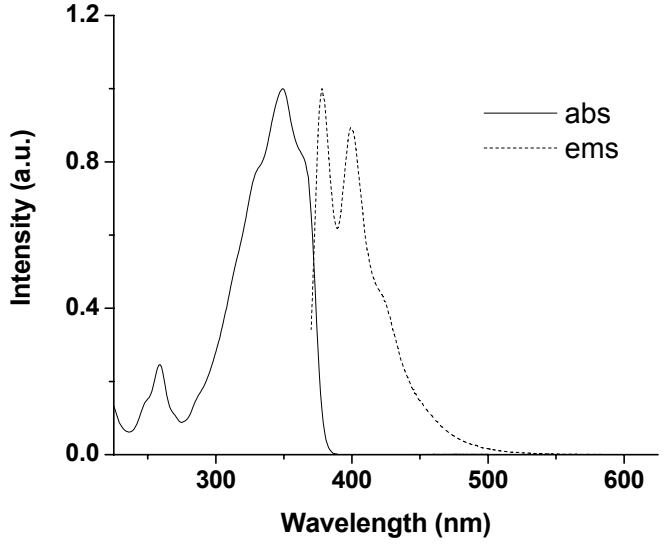


Figure S.2 Normalized Absorption and Emission Spectra for **2b** in THF.

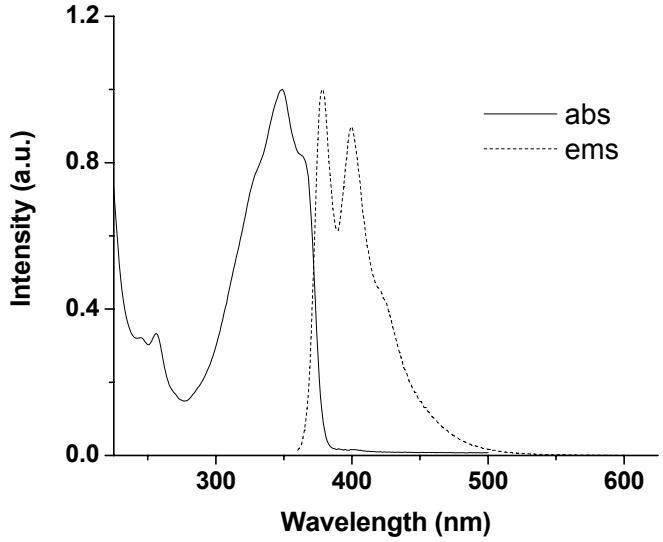


Figure S.3 Normalized Absorption and Emission Spectra for **3a** in THF.

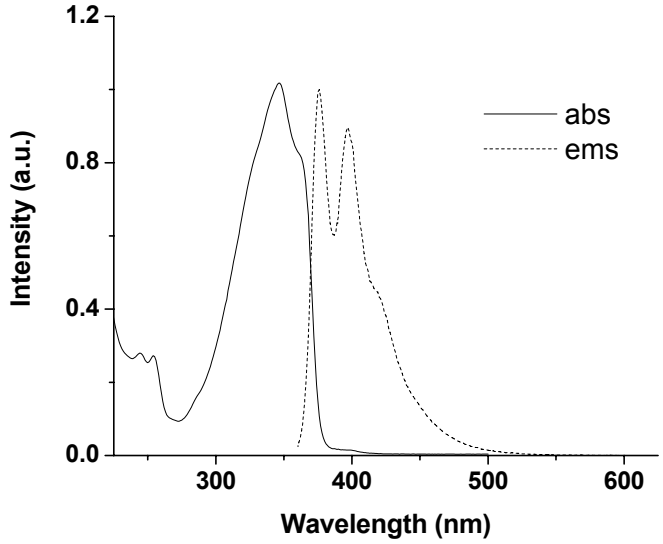


Figure S.4 Normalized Absorption and Emission Spectra for **3b** in THF.

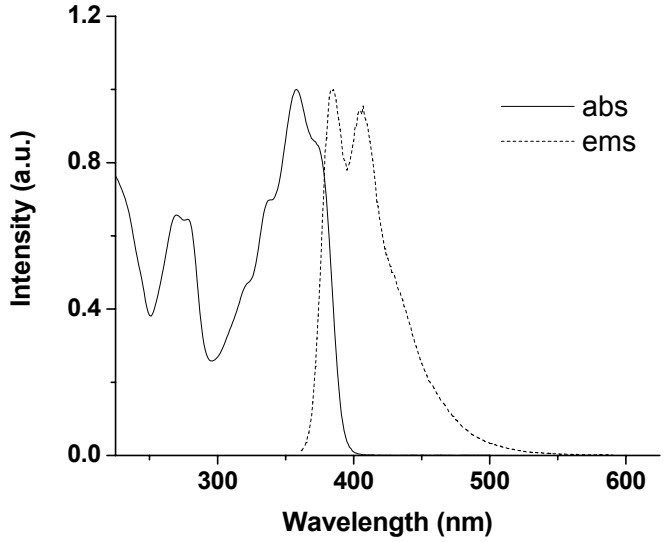


Figure S.5 Normalized Absorption and Emission Spectra for **4a** in THF.

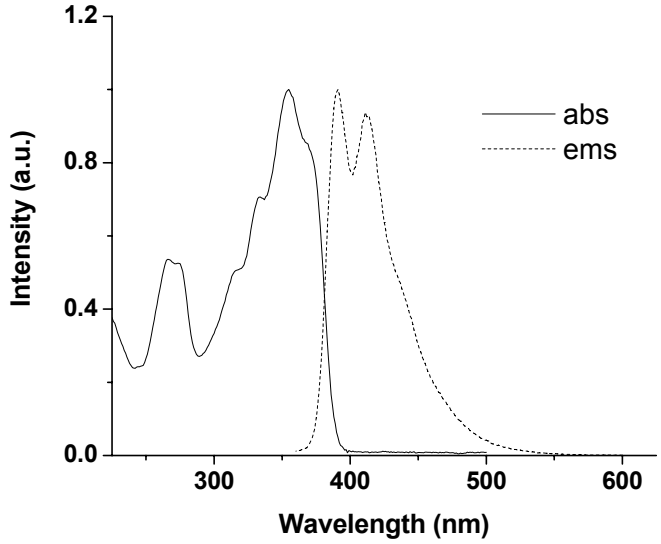


Figure S.6 Normalized Absorption and Emission Spectra for **4b** in THF.

4) Electrochemical Data

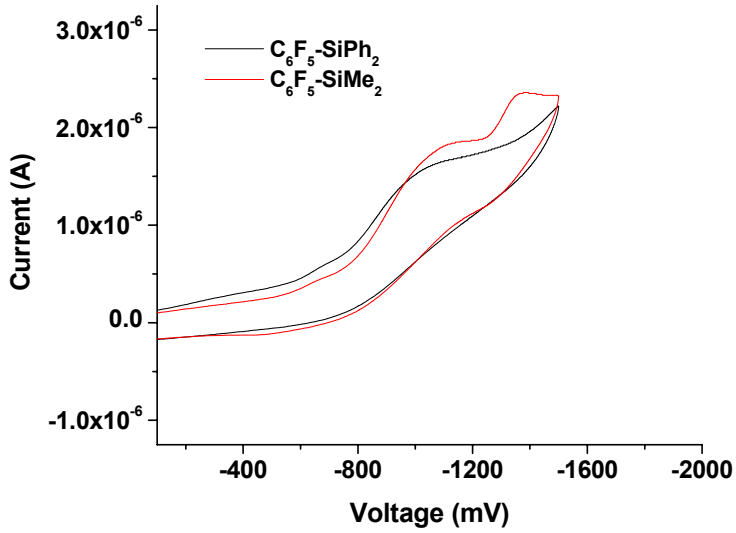


Figure S.7 Reduction behavior for **2a** (black)**, 2b** (red) in 0.1 M Bu_4NPF_6 in MeCN/PhMe, Ag wire used as reference electrode.

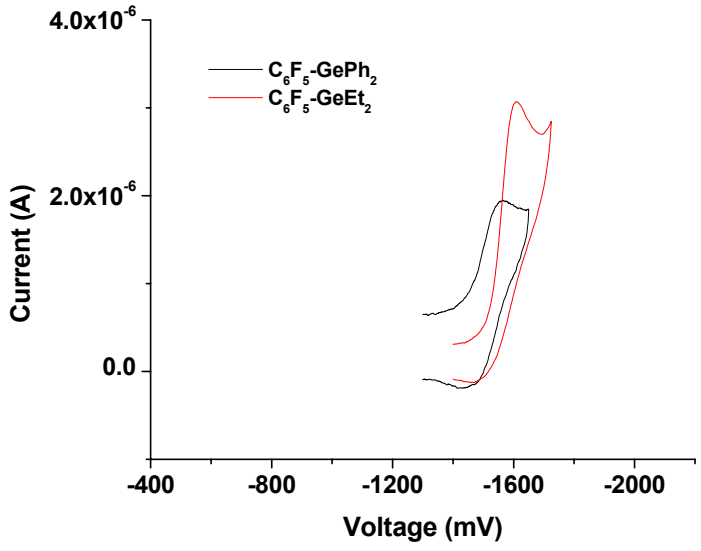


Figure S.8 Reduction behavior for **3a** (black)**, 3b** (red) in 0.1 M Bu_4NPF_6 in MeCN.

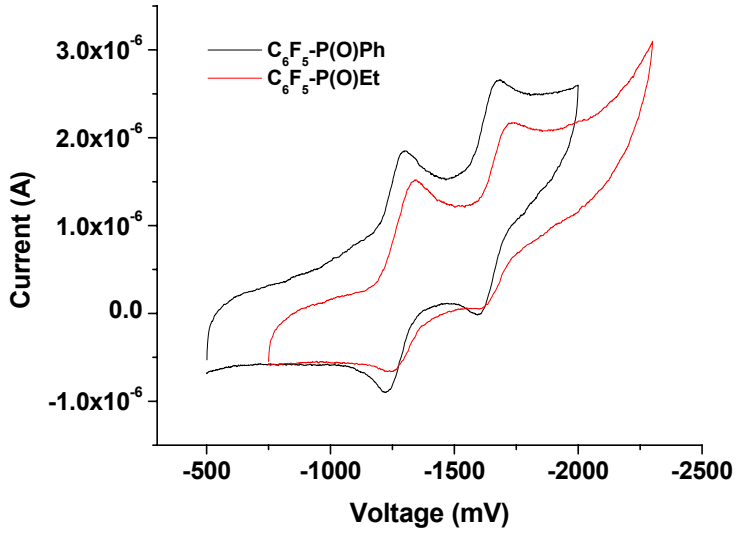


Figure S.9 Reduction behavior for **4a** (black)**, 4b** (red) in 0.1 M Bu_4NPF_6 in MeCN.

5) Additional Photo Volataic Performance Data

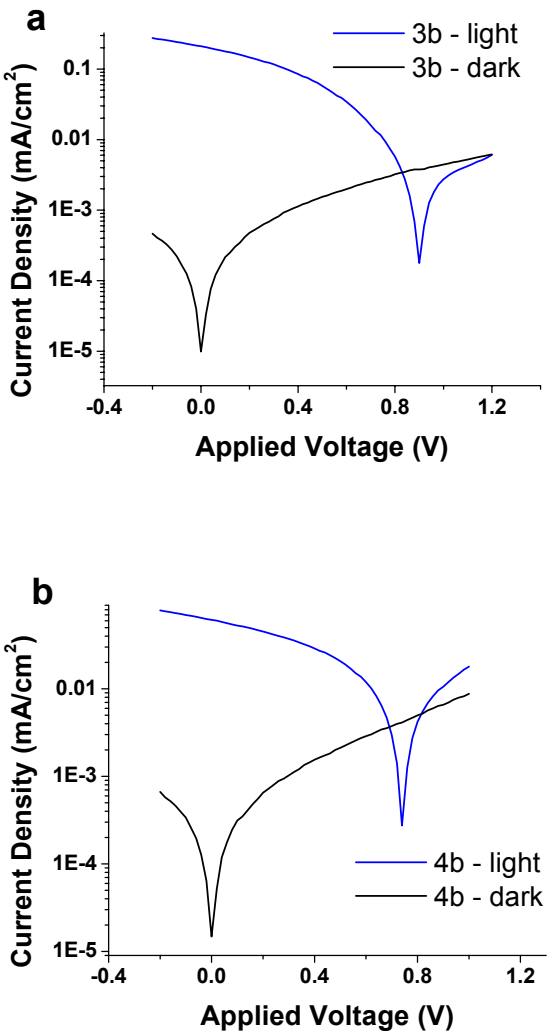


Figure S. 10 Light (blue) and dark (black) photocurrent response of spin coated devices with **3b**/P3HT blend (a) and **4b**/P3HT blend (b).

5) Crystal Structure Data

General Comments:
It is worth noting that collection out to 25 degrees was performed however, poor crystal quality resulted in no reflections past 20 degrees. Additionally, a cut-off was made in the integration to preserve the integrity of the absorption correction. The poor data:parameter ratio in these crystals can be attributed to both poor crystal quality and the lack of high angle data.

Compound 4a

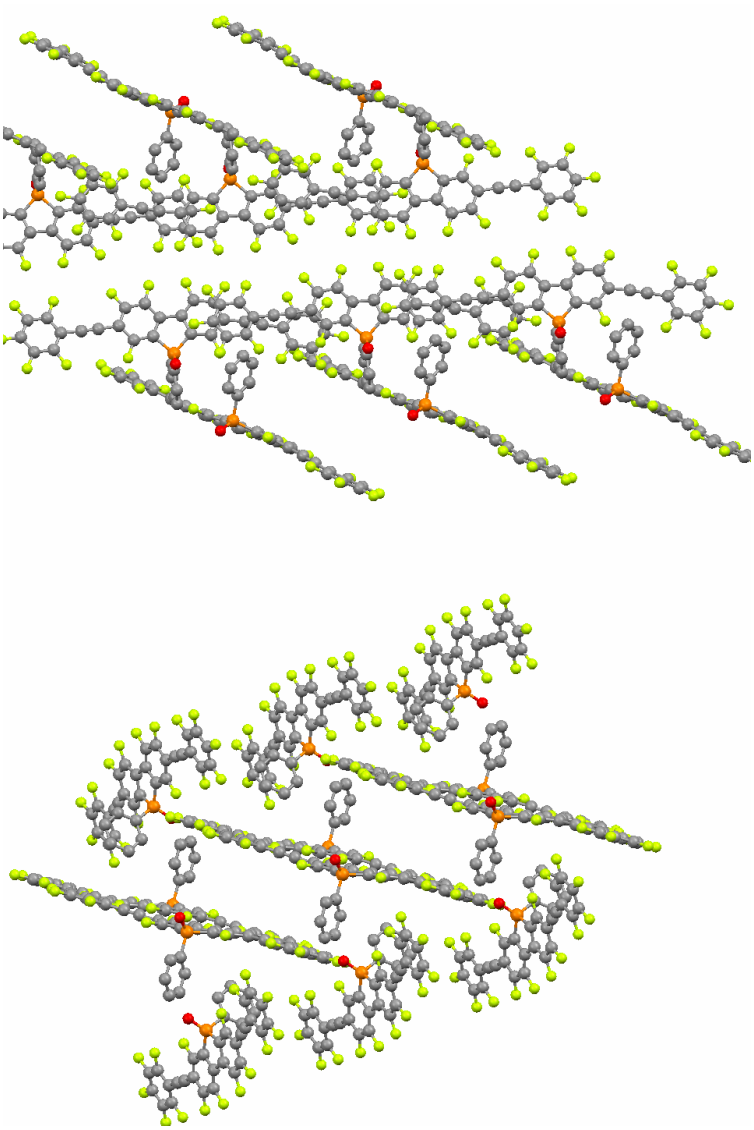


Figure S11. Crystal structures for phosphafuorene **4a**, illustrating the bi-directional nature of the crystal packing. Hydrogen atoms have been removed for clarity.

Data Collection

A fragment of a colorless needle-like crystal of C34 H5 F16 O P having approximate dimensions of 0.2 x 0.07 x 0.07 mm was mounted on a glass fiber using Paratone N hydrocarbon oil. All measurements were made on a CCD area detector [10] CCD area detector with graphite monochromated MoKradiation.

Cell constants and an orientation matrix, obtained from a least-squares refinement using the measured positions of 986 centered reflections with $I > 10\sigma(I)$ in the range $2.35 < \theta < 19.08^{o}$ corresponded to a primitive Monoclinic cell with dimensions:

a = 13.798(13) Å	$\alpha = 90^{o}$
b = 5.938(5) Å	$\beta = 100.599(16)^{o}$
c = 35.27(3) Å	$\gamma = 90^{o}$
V = 8876(1) Å^3	

For Z = 4 and F.W. = 764.35, the calculated density is 1.788 g/cm^3.

Analysis of the systematic absences allowed the space group to be uniquely determined to be:

P2(1)/n

The data were collected at a temperature of 140(2) K. Frames corresponding to an arbitrary hemisphere of data were collected using ω scans of 0.3^{o} counted for a total of 30 seconds per frame.

Data Reduction

Data were integrated by the program SAINT[11] to a maximum θ value of 20.86^{o}. The data were corrected for Lorentz and polarization effects. Data were analyzed for agreement and possible absorption using XPREP[12]. An empirical absorption correction based on comparison of redundant and equivalent reflections was applied using SADABS[13]. (Tmax = 0.990, Tmin = 0.981). Of the 9224 reflections that were collected, 2955 were unique (R_{int} = 0.0957); equivalent reflections were merged. No decay correction was applied.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 2955 reflections (all data) and 469 variable parameters and converged (largest parameter shift was 0.001 times its esd) with conventional unweighted and weighted agreement factors of:

$$R_1 = \Sigma||Fo| - |Fc|| / \Sigma|Fo| = 0.0374 \text{ for } 1603 \text{ data with } I > 2\sigma(I)$$

$$wR_2 = [(\Sigma w (|Fo|^2 - |Fc|^2)^2 / \Sigma w |Fo|^2)]^{1/2} = 0.0420$$

The standard deviation of an observation of unit weight[4] was 0.798. The weighting scheme was based on counting statistics and included a factor to down weight the intense reflections. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.236 and -0.280 e$^-$/Å^3, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the SHELXTL[9] crystallographic software package of Bruker Analytical X-ray Systems Inc.

References

(1) XS: Program for the Solution of X-ray Crystal Structures, Part of the SHELXTL Crystal Structure Determination Package, Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(2) XL: Program for the Refinement of X-ray Crystal Structures, Part of the SHELXTL Crystal Structure Determination Package, Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(3) Least-Squares:

Function minimized $\Sigma w\ (|Fo|^2 - |Fc|^2)^2$

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(|Fo|^2 - |Fc|^2)^2/(N_o - N_v)]^{1/2}$
where N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) XP: Molecular Graphics program. Part of the SHELXTL Structure Determination Package. Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(10)SMART: Area-Detector Software Package, Bruker Analytical X-ray Systems, Inc.: Madison, WI, (1995-99)

(11)SAINT: SAX Area-Dectector Integration Program, V5.04; Siemens Industrial Automation, Inc.: Madison, WI, (1995)

(12)XPREP:(v 5.03) Part of the SHELXTL Crystal Structure Determination Package, Siemens Industrial Automation, Inc.: Madison, WI, (1995)

(13)SADABS: Siemens Area Detector ABSorption correction program, George Sheldrick, (1996). Advance copy, private communication.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C34 H5 F16 O P
Formula Weight	764.35
Crystal Color, Habit	colorless, needle
Crystal Dimensions	0.2 x 0.07 x 0.07 mm
Crystal System	Monoclinic
Lattice Type	primitive
Lattice Parameters	a = 13.798(13) Å b = 5.938(5) Å c = 35.27(3) Å α= 90 ^{o} β= 100.599(16) ^{o} γ = 90^{o} V = 2840(5) Å^3
Space Group	P2(1)/n
Z value	4
D_{calc}	1.788 g/cm^3
F_{000}	1504
μ(MoK)	0.23 cm^{-1}

B. Intensity Measurements

Diffractometer	CCD area detector
Radiation	MoK(λ = 0.71073 Å) graphite monochromated
Detector Position	60.00 mm
Exposure Time	30 seconds per frame.
Scan Type	ω (0.3 degrees per frame)
θ_{max}	20.86^{o}
No. of Reflections Measured	Total: 9224 Unique: 2955 (R_{int} = 0.0957)
Corrections	Lorentz-polarization Absorption (Tmax = 0.990, Tmin = 0.981)

C. Structure Solution and Refinement

Structure Solution	direct (SHELXS-97 (Sheldrick, 1990))
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|F_o\|^2 - \|F_c\|^2)^2$
Least Squares Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (qP)^2 + 0.000P]$ where $P = [F_o^2 + 2F_c^2]/3$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	1603
No. Variables	469
Residuals: R; wR_2; Rall	0.0374; 0.0420; 0.0909
Goodness of Fit Indicator	0.798
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.236 e$^-$/Å^3
Minimum peak in Final Diff. Map	-0.280 e$^-$/Å^3

Table 1. Atomic coordinates and U_{iso}/U_{eq} and occupancy

atom	x	y	z	U_{eq}	Occupancy

P1	0.7589(1)	0.4211(2)	0.1473(1)	0.034(1)	1
F5	1.2203(2)	0.4800(4)	0.0050(1)	0.047(1)	1
F11	0.5573(2)	0.2742(4)	0.1666(1)	0.043(1)	1
F7	0.9869(2)	0.1405(4)	0.0303(1)	0.045(1)	1
F6	0.9459(2)	0.6563(4)	0.1268(1)	0.043(1)	1
F12	0.3039(2)	0.1203(4)	0.2066(1)	0.051(1)	1
F10	0.5121(2)	-0.3335(4)	0.0801(1)	0.044(1)	1
F4	1.3756(2)	0.7017(4)	-0.0131(1)	0.051(1)	1
F2	1.3354(2)	1.2813(4)	0.0730(1)	0.047(1)	1
F1	1.1788(2)	1.0590(4)	0.0922(1)	0.047(1)	1
F8	0.8349(2)	-0.0942(4)	0.0416(1)	0.044(1)	1
F9	0.6735(2)	-0.2337(4)	0.0552(1)	0.045(1)	1
F13	0.1479(2)	-0.0003(4)	0.2383(1)	0.054(1)	1
F14	0.0532(2)	-0.3960(4)	0.2172(1)	0.057(1)	1
O1	0.7239(2)	0.6551(4)	0.1489(1)	0.038(1)	1
F16	0.2742(2)	-0.5528(4)	0.1351(1)	0.052(1)	1
F15	0.1155(2)	-0.6687(4)	0.1654(1)	0.052(1)	1
F3	1.4325(2)	1.1028(4)	0.0212(1)	0.049(1)	1
C34	0.2926(4)	-0.2131(8)	0.1695(1)	0.033(1)	1
C19	0.8556(4)	0.0928(7)	0.0631(1)	0.028(1)	1
C18	0.8000(4)	0.1656(7)	0.0896(1)	0.028(1)	1
C7	1.1965(3)	0.7632(7)	0.0494(1)	0.027(1)	1
C17	0.8354(3)	0.3555(7)	0.1117(1)	0.028(1)	1
C9	1.3052(4)	1.0841(8)	0.0567(1)	0.032(1)	1
C15	0.9691(3)	0.4097(8)	0.0774(1)	0.030(1)	1
C26	0.7055(3)	0.0782(7)	0.0979(1)	0.027(1)	1
C10	1.3553(4)	0.9924(8)	0.0297(2)	0.032(1)	1
C12	1.2479(4)	0.6779(8)	0.0223(1)	0.031(1)	1
C1	0.8213(3)	0.3201(7)	0.1930(1)	0.029(1)	1
C25	0.6479(4)	-0.0973(8)	0.0825(1)	0.033(1)	1
C33	0.2434(4)	-0.4135(8)	0.1604(1)	0.034(1)	1
C23	0.5288(4)	-0.0300(8)	0.1238(1)	0.032(1)	1
C21	0.6737(3)	0.2051(7)	0.1270(1)	0.024(1)	1
C6	0.8633(3)	0.1098(7)	0.1988(1)	0.035(1)	1
C30	0.1795(4)	-0.1336(8)	0.2118(1)	0.040(1)	1
C20	0.9382(4)	0.2146(8)	0.0576(1)	0.033(1)	1
C24	0.5621(4)	-0.1503(8)	0.0949(2)	0.033(1)	1
C31	0.1313(4)	-0.3362(9)	0.2017(2)	0.041(2)	1
C2	0.8248(3)	0.4621(7)	0.2240(1)	0.036(1)	1
C32	0.1632(4)	-0.4739(8)	0.1756(2)	0.035(1)	1
C11	1.3266(4)	0.7892(9)	0.0133(1)	0.037(1)	1
C14	1.0506(4)	0.5385(8)	0.0686(1)	0.038(2)	1
C29	0.2584(4)	-0.0761(8)	0.1960(1)	0.033(1)	1
C8	1.2263(4)	0.9700(8)	0.0655(1)	0.031(1)	1
C16	0.9152(4)	0.4708(7)	0.1059(1)	0.032(1)	1
C28	0.3749(4)	-0.1496(8)	0.1529(2)	0.039(2)	1
C4	0.9151(3)	0.1877(9)	0.2657(1)	0.046(2)	1
C3	0.8714(3)	0.3957(8)	0.2605(1)	0.048(2)	1
C13	1.1166(4)	0.6429(8)	0.0600(1)	0.038(2)	1
C27	0.4426(4)	-0.0975(8)	0.1387(2)	0.037(2)	1
C22	0.5882(4)	0.1521(7)	0.1380(1)	0.032(1)	1
C5	0.9108(3)	0.0437(7)	0.2349(2)	0.045(2)	1

U_{eq} is defined as one third of the orthogonalized U_{ij} tensor

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P1	0.037(1)	0.030(1)	0.034(1)	0.002(1)	0.001(1)	0.000(1)
F5	0.060(2)	0.041(2)	0.039(2)	-0.007(1)	0.004(2)	-0.007(1)
F11	0.041(2)	0.046(2)	0.042(2)	0.001(2)	0.010(2)	-0.002(1)
F7	0.045(2)	0.054(2)	0.037(2)	-0.006(1)	0.012(2)	-0.004(1)
F6	0.051(2)	0.035(2)	0.046(2)	-0.006(1)	0.011(2)	-0.013(1)
F12	0.051(2)	0.038(2)	0.062(2)	-0.007(1)	0.010(2)	-0.011(2)
F10	0.041(2)	0.049(2)	0.043(2)	-0.004(1)	0.005(1)	-0.013(1)
F4	0.051(2)	0.062(2)	0.044(2)	-0.004(2)	0.017(2)	-0.005(2)
F2	0.055(2)	0.036(2)	0.048(2)	-0.005(1)	0.002(2)	-0.012(1)
F1	0.050(2)	0.047(2)	0.045(2)	-0.009(2)	0.014(2)	-0.005(2)
F8	0.044(2)	0.038(2)	0.047(2)	-0.011(1)	0.005(2)	-0.004(1)
F9	0.046(2)	0.046(2)	0.044(2)	-0.011(2)	0.009(2)	-0.014(1)
F13	0.051(2)	0.057(2)	0.059(2)	-0.009(2)	0.022(2)	0.000(1)
F14	0.050(2)	0.056(2)	0.069(2)	-0.004(2)	0.022(2)	-0.007(2)
O1	0.045(2)	0.026(2)	0.040(2)	0.003(2)	0.005(2)	0.008(2)
F16	0.050(2)	0.052(2)	0.055(2)	-0.013(2)	0.011(2)	-0.007(2)
F15	0.046(2)	0.043(2)	0.069(2)	-0.011(2)	0.011(2)	-0.017(1)
F3	0.043(2)	0.061(2)	0.046(2)	0.002(2)	0.011(2)	-0.011(2)
C34	0.030(4)	0.029(4)	0.037(4)	0.000(3)	0.002(3)	-0.001(3)
C19	0.028(4)	0.029(3)	0.026(4)	-0.007(3)	-0.001(3)	-0.008(3)
C18	0.036(4)	0.024(3)	0.022(3)	0.001(3)	-0.002(3)	0.003(3)
C7	0.023(4)	0.019(3)	0.038(4)	0.007(3)	0.001(3)	-0.008(3)
C17	0.039(4)	0.027(3)	0.017(3)	0.002(3)	0.002(3)	-0.001(3)
C9	0.037(4)	0.024(3)	0.034(4)	0.003(3)	0.000(3)	-0.005(3)
C15	0.027(4)	0.032(3)	0.027(4)	0.003(3)	-0.001(3)	-0.008(3)
C26	0.027(4)	0.032(3)	0.022(3)	0.006(3)	0.005(3)	0.000(3)
C10	0.026(4)	0.036(4)	0.032(4)	0.010(3)	0.001(3)	-0.014(3)
C12	0.036(4)	0.025(3)	0.033(4)	-0.005(3)	0.007(3)	-0.003(3)
C1	0.034(3)	0.023(3)	0.029(3)	-0.005(3)	0.006(3)	-0.003(2)
C25	0.033(4)	0.030(3)	0.032(4)	-0.006(3)	-0.002(3)	-0.006(3)
C33	0.038(4)	0.037(4)	0.028(4)	-0.009(3)	0.008(3)	0.005(3)
C23	0.031(4)	0.033(4)	0.030(4)	0.005(3)	0.001(3)	-0.002(3)
C21	0.019(3)	0.029(3)	0.025(4)	0.007(3)	0.006(3)	-0.005(3)
C6	0.046(3)	0.031(3)	0.030(4)	-0.004(3)	0.010(3)	-0.001(3)
C30	0.048(4)	0.037(4)	0.036(4)	-0.011(3)	0.010(3)	0.004(3)
C20	0.036(4)	0.038(4)	0.025(4)	0.000(3)	0.003(3)	0.004(3)
C24	0.030(4)	0.028(3)	0.035(4)	0.004(3)	-0.008(3)	-0.016(3)
C31	0.026(4)	0.047(4)	0.052(4)	0.005(3)	0.011(3)	-0.005(3)
C2	0.039(4)	0.032(3)	0.038(4)	0.002(3)	0.007(3)	0.003(2)
C32	0.035(4)	0.028(4)	0.043(4)	-0.009(3)	0.006(3)	-0.003(3)
C11	0.045(4)	0.047(4)	0.021(4)	0.000(3)	0.013(3)	0.000(3)
C14	0.045(5)	0.042(4)	0.025(4)	0.004(3)	0.001(3)	0.005(3)
C29	0.033(4)	0.030(3)	0.036(4)	0.004(3)	0.005(3)	-0.009(3)
C8	0.034(4)	0.039(4)	0.022(4)	0.002(3)	0.011(3)	0.002(3)
C16	0.046(4)	0.019(3)	0.026(4)	-0.005(3)	-0.007(3)	-0.005(3)
C28	0.041(5)	0.032(3)	0.037(4)	0.006(3)	-0.011(3)	0.001(3)
C4	0.049(4)	0.048(4)	0.035(4)	0.007(3)	-0.011(3)	-0.007(3)
C3	0.067(4)	0.048(4)	0.027(4)	-0.016(3)	0.006(3)	-0.007(3)
C13	0.041(4)	0.040(4)	0.030(4)	0.002(3)	0.002(3)	0.000(3)
C27	0.036(5)	0.030(3)	0.040(4)	-0.002(3)	-0.010(3)	-0.005(3)
C22	0.046(4)	0.031(4)	0.018(3)	-0.003(3)	0.000(3)	0.008(3)
C5	0.053(4)	0.031(3)	0.046(4)	0.006(3)	-0.004(3)	0.002(3)

The general temperature factor expression:
$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
P1	O1	1.476(3)	P1	C1	1.785(4)
P1	C21	1.797(4)	P1	C17	1.825(5)
F5	C12	1.346(5)	F11	C22	1.372(5)
F7	C20	1.346(5)	F6	C16	1.349(4)
F12	C29	1.344(4)	F10	C24	1.342(4)
F4	C11	1.352(5)	F2	C9	1.335(5)
F1	C8	1.349(5)	F8	C19	1.346(4)
F9	C25	1.352(5)	F13	C30	1.357(5)
F14	C31	1.345(5)	F16	C33	1.342(5)
F15	C32	1.347(5)	F3	C10	1.333(5)
C34	C33	1.378(6)	C34	C29	1.386(6)
C34	C28	1.421(6)	C19	C18	1.382(6)
C19	C20	1.393(5)	C18	C17	1.406(5)
C18	C26	1.481(6)	C7	C8	1.384(5)
C7	C12	1.386(6)	C7	C13	1.421(6)
C17	C16	1.344(5)	C9	C8	1.365(6)
C9	C10	1.387(6)	C15	C20	1.380(5)
C15	C16	1.402(6)	C15	C14	1.441(6)
C26	C25	1.362(5)	C26	C21	1.410(5)
C10	C11	1.365(6)	C12	C11	1.357(6)
C1	C6	1.376(5)	C1	C2	1.376(5)
C25	C24	1.373(6)	C33	C32	1.362(6)
C23	C24	1.389(6)	C23	C22	1.393(6)
C23	C27	1.442(6)	C21	C22	1.344(6)
C6	C5	1.375(5)	C6	H6A	0.9300
C30	C29	1.354(6)	C30	C31	1.389(6)
C31	C32	1.363(6)	C2	C3	1.384(5)
C2	H2A	0.9300	C14	C13	1.186(6)
C28	C27	1.180(6)	C4	C3	1.372(5)
C4	C5	1.376(5)	C4	H4A	0.9300
C3	H3A	0.9300	C5	H5A	0.9300

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
O1	P1	C1	112.9(2)	O1	P1	C21	119.47(19)
C1	P1	C21	106.89(19)	O1	P1	C17	117.51(19)
C1	P1	C17	107.8(2)	C21	P1	C17	89.6(2)
C33	C34	C29	116.7(5)	C33	C34	C28	122.0(5)
C29	C34	C28	121.3(5)	F8	C19	C18	123.6(4)
F8	C19	C20	116.4(5)	C18	C19	C20	120.1(5)
C19	C18	C17	116.5(5)	C19	C18	C26	129.9(5)
C17	C18	C26	113.6(5)	C8	C7	C1	117.2(5)
C8	C7	C13	121.6(5)	C12	C7	C13	121.2(5)
C16	C17	C18	122.3(5)	C16	C17	P1	126.2(4)
C18	C17	P1	111.5(4)	F2	C9	C8	122.4(5)
F2	C9	C10	118.9(5)	C8	C9	C10	118.7(5)
C20	C15	C16	115.1(5)	C20	C15	C14	121.1(5)
C16	C15	C14	123.8(5)	C25	C26	C21	117.5(5)
C25	C26	C18	131.3(5)	C21	C26	C18	111.1(4)
F3	C10	C11	121.4(5)	F3	C10	C9	118.9(5)
C11	C10	C9	119.7(5)	F5	C12	C11	119.5(5)
F5	C12	C7	119.5(5)	C11	C12	C7	121.0(5)
C6	C1	C2	119.0(4)	C6	C1	P1	124.1(4)
C2	C1	P1	116.9(3)	F9	C25	C26	122.1(5)
F9	C25	C24	116.3(4)	C26	C25	C24	121.5(5)
F16	C33	C32	118.5(5)	F16	C33	C34	119.0(5)
C32	C33	C34	122.5(5)	C24	C23	C22	114.2(5)
C24	C23	C27	122.0(5)	C22	C23	C27	123.7(5)
C22	C21	C26	119.5(4)	C22	C21	P1	126.7(4)
C26	C21	P1	113.7(4)	C5	C6	C1	120.9(4)
C5	C6	H6A	119.5	C1	C6	H6A	119.5
C29	C30	F13	121.4(5)	C29	C30	C31	119.7(5)
F13	C30	C31	118.9(5)	F7	C20	C15	119.2(5)
F7	C20	C19	117.3(5)	C15	C20	C19	123.4(5)
F10	C24	C25	118.2(5)	F10	C24	C23	119.1(5)
C25	C24	C23	122.5(5)	F14	C31	C32	120.0(5)
F14	C31	C30	120.4(5)	C32	C31	C30	119.6(5)
C1	C2	C3	120.5(4)	C1	C2	H2A	119.8
C3	C2	H2A	119.8	F15	C32	C33	120.9(5)
F15	C32	C31	119.5(5)	C33	C32	C31	119.5(5)
F4	C11	C12	119.8(5)	F4	C11	C10	119.2(5)
C12	C11	C10	120.9(5)	C13	C14	C15	177.8(5)
F12	C29	C30	118.3(5)	F12	C29	C34	119.8(5)
C30	C29	C34	121.9(5)	F1	C8	C9	118.3(5)
F1	C8	C7	119.3(5)	C9	C8	C7	122.4(5)
C17	C16	F6	121.1(5)	C17	C16	C15	122.4(4)
F6	C16	C15	116.6(5)	C27	C28	C34	179.2(6)
C3	C4	C5	119.9(4)	C3	C4	H4A	120.0
C5	C4	H4A	120.0	C4	C3	C2	119.9(4)
C4	C3	H3A	120.1	C2	C3	H3A	120.1
C14	C13	C7	178.6(6)	C28	C27	C23	176.3(6)
C21	C22	F11	119.7(4)	C21	C22	C23	124.6(5)
F11	C22	C23	115.6(5)	C6	C5	C4	119.8(4)
C6	C5	H5A	120.1	C4	C5	H5A	120.1

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
F8	C19	C18	C17	175.8(4)	C20	C19	C18	C17	-4.1(6)
F8	C19	C18	C26	-6.4(7)	C20	C19	C18	C26	173.7(4)
C19	C18	C17	C16	4.3(6)	C26	C18	C17	C16	173.9(4)
C19	C18	C17	P1	-176.5(3)	C26	C18	C17	P1	5.4(4)
O1	P1	C17	C16	49.7(4)	C1	P1	C17	C16	-79.2(4)
C21	P1	C17	C16	173.2(4)	O1	P1	C17	C18	-129.5(3)
C1	P1	C17	C18	101.6(3)	C21	P1	C17	C18	-6.0(3)
C19	C18	C26	C25	1.1(8)	C17	C18	C26	C25	178.9(4)
C19	C18	C26	C21	-179.2(4)	C17	C18	C26	C21	-1.4(5)
F2	C9	C10	F3	0.8(6)	C8	C9	C10	F3	-179.8(4)
F2	C9	C10	C11	178.9(4)	C8	C9	C10	C11	-1.7(7)
C8	C7	C12	F5	-178.4(3)	C13	C7	C12	F5	1.7(7)
C8	C7	C12	C11	2.5(7)	C13	C7	C12	C11	-177.4(4)
O1	P1	C1	C6	-178.0(3)	C21	P1	C1	C6	48.7(4)
C17	P1	C1	C6	-46.5(4)	O1	P1	C1	C2	3.4(4)
C21	P1	C1	C2	-129.9(4)	C17	P1	C1	C2	134.9(4)
C21	C26	C25	F9	-177.8(4)	C18	C26	C25	F9	1.9(7)
C21	C26	C25	C24	-0.3(6)	C18	C26	C25	C24	179.4(4)
C29	C34	C33	F16	-179.5(4)	C28	C34	C33	F16	-0.1(6)
C29	C34	C33	C32	2.0(7)	C28	C34	C33	C32	-178.6(4)
C25	C26	C21	C22	-1.2(6)	C18	C26	C21	C22	179.0(4)
C25	C26	C21	P1	176.4(3)	C18	C26	C21	P1	-3.4(4)
O1	P1	C21	C22	-55.5(4)	C1	P1	C21	C22	74.2(4)
C17	P1	C21	C22	-177.3(4)	O1	P1	C21	C26	127.2(3)
C1	P1	C21	C26	-103.2(3)	C17	P1	C21	C26	5.4(3)
C2	C1	C6	C5	-1.9(7)	P1	C1	C6	C5	179.6(3)
C16	C15	C20	F7	-178.7(4)	C14	C15	C20	F7	1.9(6)
C16	C15	C20	C19	4.1(7)	C14	C15	C20	C19	-175.2(4)
F8	C19	C20	F7	2.8(6)	C18	C19	C20	F7	-177.3(4)
F8	C19	C20	C15	180.0(4)	C18	C19	C20	C15	-0.1(7)
F9	C25	C24	F10	1.5(6)	C26	C25	C24	F10	-176.1(4)
F9	C25	C24	C23	177.5(4)	C26	C25	C24	C23	-0.1(7)
C22	C23	C24	F10	177.9(3)	C27	C23	C24	F10	0.8(7)
C22	C23	C24	C25	1.9(7)	C27	C23	C24	C25	-175.2(4)
C29	C30	C31	F14	179.6(4)	F13	C30	C31	F14	1.7(7)
C29	C30	C31	C32	-0.9(7)	F13	C30	C31	C32	-178.8(4)
C6	C1	C2	C3	1.0(7)	P1	C1	C2	C3	179.7(3)
F16	C33	C32	F15	-0.1(7)	C34	C33	C32	F15	178.4(4)
F16	C33	C32	C31	179.3(4)	C34	C33	C32	C31	-2.2(7)
F14	C31	C32	F15	0.5(7)	C30	C31	C32	F15	-179.0(4)
F14	C31	C32	C33	-178.9(4)	C30	C31	C32	C33	1.6(7)
F5	C12	C11	F4	0.7(7)	C7	C12	C11	F4	179.8(4)
F5	C12	C11	C10	178.6(4)	C7	C12	C11	C10	-2.3(7)
F3	C10	C11	F4	-2.2(7)	C9	C10	C11	F4	179.8(4)
F3	C10	C11	C12	179.9(4)	C9	C10	C11	C12	1.8(7)
C20	C15	C14	C13	38(15)	C16	C15	C14	C13	-141(15)
F13	C30	C29	F12	-1.7(7)	C31	C30	C29	F12	-179.6(4)
F13	C30	C29	C34	178.7(4)	C31	C30	C29	C34	0.7(7)
C33	C34	C29	F12	179.0(4)	C28	C34	C29	F12	-0.4(6)
C33	C34	C29	C30	-1.3(7)	C28	C34	C29	C30	179.3(4)
F2	C9	C8	F1	-1.4(6)	C10	C9	C8	F1	179.2(4)
F2	C9	C8	C7	-178.6(4)	C10	C9	C8	C7	2.1(7)
C12	C7	C8	F1	-179.6(4)	C13	C7	C8	F1	0.3(6)
C12	C7	C8	C9	-2.4(7)	C13	C7	C8	C9	177.5(4)

C18	C17	C16	F6	178.2(3)	P1	C17	C16	F6 -1.0(6)
C18	C17	C16	C15	-0.2(7)	P1	C17	C16	C15 -179.3(3)
C20	C15	C16	C17	-4.0(7)	C14	C15	C16	C17 175.4(4)
C20	C15	C16	F6	177.6(3)	C14	C15	C16	F6 -3.0(6)
C33	C34	C28	C27	47(44)	C29	C34	C28	C27 -134(44)
C5	C4	C3	C2	-1.4(7)	C1	C2	C3	C4 0.6(7)
C15	C14	C13	C7	-60(31)	C8	C7	C13	C14 -163(100)
C12	C7	C13	C14	17(24)	C34	C28	C27	C23 -180(100)
C24	C23	C27	C28	140(8)	C22	C23	C27	C28 -37(9)
C26	C21	C22	F11	179.5(4)	P1	C21	C22	F11 2.3(6)
C26	C21	C22	C23	3.3(7)	P1	C21	C22	C23 -173.9(3)
C24	C23	C22	C21	-3.6(7)	C27	C23	C22	C21 173.5(4)
C24	C23	C22	F11	-179.9(4)	C27	C23	C22	F11 -2.9(6)
C1	C6	C5	C4	1.1(7)	C3	C4	C5	C6 0.6(7)

Compound 3a

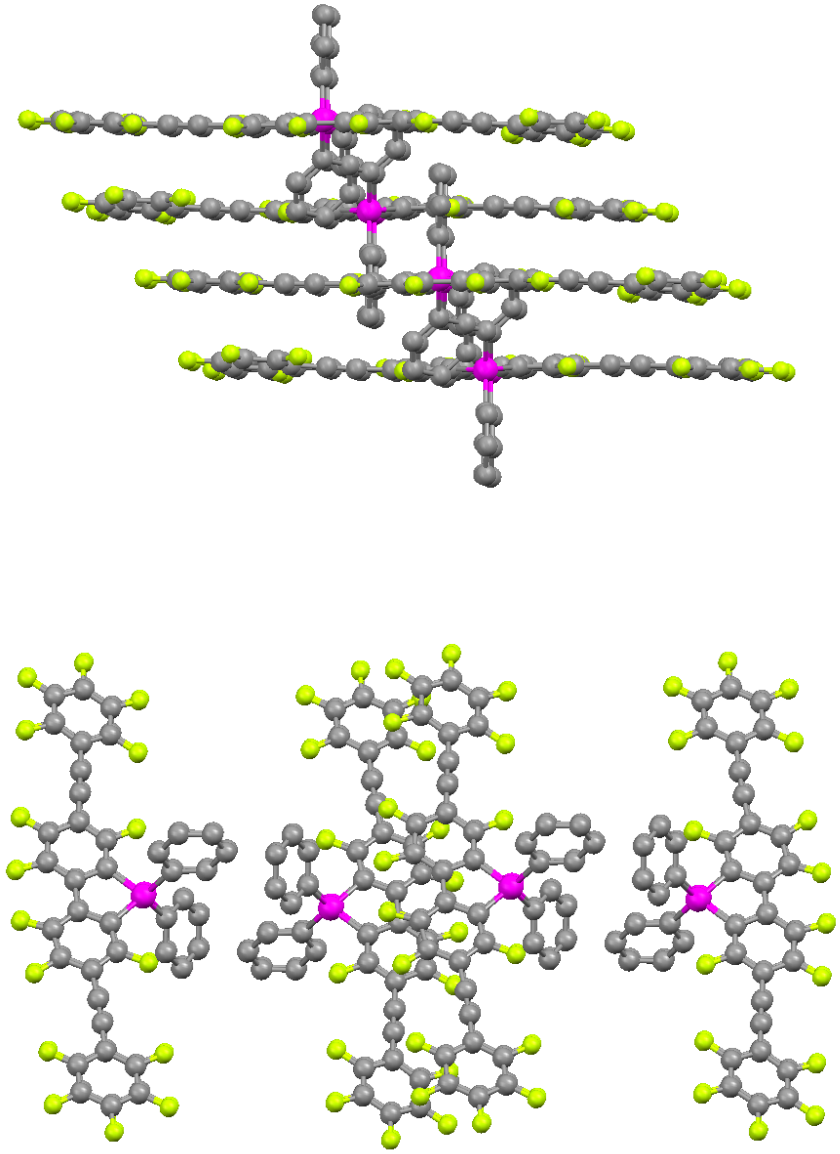


Figure S12. Additional views for the Crystal structure for germafluorene **3a**, illustrating the bi-directional nature of the crystal packing. Hydrogen atoms have been removed for clarity.

Data Collection

A fragment of a colorless block -like crystal of C40 H10 F16 Ge having approximate dimensions of .17 x .07 x .05 mm was mounted on a glass fiber using Paratone N hydrocarbon oil. All measurements were made on a CCD area detector [10] CCD area detector with graphite monochromated MoKradiation.

Cell constants and an orientation matrix, obtained from a least-squares refinement using the measured positions of 1247 centered reflections with $I > 10\sigma(I)$ in the range $2.52 < \theta < 19.89^{o}$ corresponded to a primitive Triclinic cell with dimensions:

a = 7.922(2) Å	α = 111.63(3) ^{o}
b = 15.049(3) Å	β = 102.29(3) ^{o}
c = 15.823(3) Å	γ = 101.99(3) ^{o}
V = 8876(1) Å^3	

For Z = 2 and F.W. = 867.07, the calculated density is 1.771 g/cm^3.

Analysis of the systematic absences allowed the space group to be uniquely determined to be:

P-1

The data were collected at a temperature of 118(2) K. Frames corresponding to an arbitrary hemisphere of data were collected using ω scans of 0.3^{o} counted for a total of 30 seconds per frame.

Data Reduction

Data were integrated by the program SAINT[11] to a maximum θ value of 20.81^{o}. The data were corrected for Lorentz and polarization effects. Data were analyzed for agreement and possible absorption using XPREP[12]. An empirical absorption correction based on comparison of redundant and equivalent reflections was applied using SADABS[13]. (Tmax = 0.950, Tmin = 0.910). Of the 9156 reflections that were collected, 3384 were unique (R_{int} = 0.1023); equivalent reflections were merged. No decay correction was applied.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. Non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 3384 reflections (all data) and 514 variable parameters and converged (largest parameter shift was 0.001 times its esd) with conventional unweighted and weighted agreement factors of:

$$R_1 = \Sigma||Fo| - |Fc|| / \Sigma|Fo| = 0.0344 \text{ for 1788 data with } I > 2\sigma(I)$$

$$wR_2 = [(\Sigma w (|Fo|^2 - |Fc|^2)^{\,2} / \Sigma w |Fo|^{\,2})]^{\,1/2} = 0.0545$$

The standard deviation of an observation of unit weight[4] was 0.543. The weighting scheme was based on counting statistics and included a factor to downweight the intense reflections. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.358 and -0.256 e$^-$/Å^3, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the SHELXTL[9] crystallographic software package of Bruker Analytical X-ray Systems Inc.

References

(1) XS: Program for the Solution of X-ray Crystal Structures, Part of the SHELXTL Crystal Structure Determination Package, Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(2) XL: Program for the Refinement of X-ray Crystal Structures, Part of the SHELXTL Crystal Structure Determination Package, Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(3) Least-Squares:

Function minimized $\Sigma w\ (|Fo|^2 - |Fc|^2)^2$

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(|Fo|^2 - |Fc|^2)^2/(N_o - N_v)]^{1/2}$
where N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) XP: Molecular Graphics program. Part of the SHELXTL Structure Determination Package. Bruker Analytical X-ray Systems Inc.: Madison, WI, (1995-99)

(10)SMART: Area-Detector Software Package, Bruker Analytical X-ray Systems, Inc.: Madison, WI, (1995-99)

(11)SAINT: SAX Area-Dectector Integration Program, V5.04; Siemens Industrial Automation, Inc.: Madison, WI, (1995)

(12)XPREP:(v 5.03) Part of the SHELXTL Crystal Structure Determination Package, Siemens Industrial Automation, Inc.: Madison, WI, (1995)

(13)SADABS: Siemens Area Detector ABSorption correction program, George Sheldrick, (1996). Advance copy, private communication.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula		C40 H10 F16 Ge
Formula Weight		867.07
Crystal Color, Habit		colorless, block
Crystal Dimensions		.17 x .07 x .05 mm
Crystal System		Triclinic
Lattice Type		primitive
Lattice Parameters		a = 7.922(2) Å b = 15.049(3) Å c = 15.823(3) Å α= 111.63(3) ^{o} β= 102.29(3) ^{o} γ = 101.99(3)^{o} V = 1626.1(8) Å^3
Space Group		P-1
Z value	2	
D_{calc}		1.771 g/cm^3
F_{000}		852
μ(MoK)		1.07 cm^{-1}

B. Intensity Measurements

Diffractometer	CCD area detector
Radiation	MoK(λ = 0.71073 Å) graphite monochromated
Detector Position	60.00 mm
Exposure Time	30 seconds per frame.
Scan Type	ω (0.3 degrees per frame)
θ_{max}	20.81^{o}
No. of Reflections Measured	Total: 9156 Unique: 3384 (R_{int} = 0.1023)
Corrections	Lorentz-polarization Absorption (Tmax = 0.950, Tmin = 0.910)

C. Structure Solution and Refinement

Structure Solution	direct (SHELXS-97 (Sheldrick, 1990))
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|F_o\|^2 - \|F_c\|^2)^2$
Least Squares Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (qP)^2 + 0.000P]$ where $P = [F_o^2 + 2F_c^2]/3$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	1788
No. Variables	514
Residuals: R; wR_2; Rall	0.0344; 0.0545; 0.0802
Goodness of Fit Indicator	0.543
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.358 e$^-$/Å^3
Minimum peak in Final Diff. Map	-0.256 e$^-$/Å^3

Table 1. Atomic coordinates and U_{iso}/U_{eq} and occupancy

atom	x	y	z	U_{eq}	Occupancy
Ge1	-0.7346(1)	-0.2349(1)	-0.4748(1)	0.035(1)	1
F1	-1.5853(4)	-0.4887(2)	-0.9267(2)	0.054(1)	1
F2	-1.8931(5)	-0.5692(2)	-1.0790(2)	0.064(1)	1
F3	-2.0270(5)	-0.4491(2)	-1.1464(2)	0.067(1)	1
F4	-1.8509(4)	-0.2469(2)	-1.0646(2)	0.055(1)	1
F5	-1.5470(4)	-0.1665(2)	-0.9125(2)	0.049(1)	1
F6	-1.0833(4)	-0.3331(2)	-0.6674(2)	0.045(1)	1
F7	-1.1011(4)	-0.0321(2)	-0.6893(2)	0.046(1)	1
F8	-0.8166(4)	0.0720(2)	-0.5391(2)	0.045(1)	1
F9	-0.5546(4)	0.1478(2)	-0.3947(2)	0.045(1)	1
F10	-0.2774(4)	0.2016(2)	-0.2441(2)	0.044(1)	1
F12	0.1273(4)	-0.0419(2)	-0.0689(2)	0.057(1)	1
F13	0.4313(4)	0.0198(3)	0.0806(2)	0.065(1)	1
F14	0.6045(5)	0.2192(3)	0.1905(2)	0.076(1)	1
F15	0.4728(4)	0.3567(2)	0.1522(2)	0.070(1)	1
F16	0.1686(4)	0.2956(2)	0.0035(2)	0.056(1)	1
C1	-0.6172(7)	-0.3259(4)	-0.5413(4)	0.032(2)	1
C2	-0.4829(9)	-0.3489(4)	-0.4907(4)	0.057(2)	1
C3	-0.3938(9)	-0.4112(5)	-0.5368(5)	0.069(2)	1
C4	-0.4340(8)	-0.4518(4)	-0.6343(5)	0.052(2)	1
C5	-0.5717(9)	-0.4307(4)	-0.6868(4)	0.048(2)	1
C6	-0.6594(8)	-0.3669(4)	-0.6397(4)	0.044(2)	1
C7	-0.8698(7)	-0.2833(4)	-0.4049(3)	0.033(2)	1
C8	-0.9567(8)	-0.2227(4)	-0.3500(4)	0.043(2)	1
C9	-1.0523(8)	-0.2545(5)	-0.2968(4)	0.047(2)	1
C10	-1.0657(8)	-0.3486(5)	-0.3002(4)	0.045(2)	1
C11	-0.9829(9)	-0.4087(5)	-0.3522(4)	0.048(2)	1
C12	-0.8861(7)	-0.3764(4)	-0.4042(4)	0.038(2)	1
C13	-1.6501(9)	-0.4259(5)	-0.9595(4)	0.042(2)	1
C14	-1.8037(9)	-0.4695(5)	-1.0371(4)	0.042(2)	1
C15	-1.8739(8)	-0.4087(5)	-1.0712(4)	0.041(2)	1
C16	-1.7862(9)	-0.3079(5)	-1.0308(4)	0.039(2)	1
C17	-1.6316(8)	-0.2655(4)	-0.9534(4)	0.032(2)	1
C18	-1.5545(8)	-0.3241(5)	-0.9147(4)	0.035(2)	1
C19	-1.3946(9)	-0.2804(4)	-0.8350(4)	0.041(2)	1
C20	-1.2609(8)	-0.2392(4)	-0.7664(4)	0.036(2)	1
C21	-1.1031(7)	-0.1862(4)	-0.6836(4)	0.033(2)	1
C22	-1.0136(8)	-0.2316(4)	-0.6328(4)	0.035(2)	1
C23	-0.8639(8)	-0.1793(4)	-0.5516(4)	0.031(2)	1
C24	-0.7894(7)	-0.0740(4)	-0.5149(4)	0.031(1)	1
C25	-0.8757(8)	-0.0275(4)	-0.5651(4)	0.031(2)	1
C26	-1.0253(8)	-0.0837(5)	-0.6462(4)	0.032(2)	1
C27	-0.6267(7)	-0.0279(4)	-0.4266(4)	0.027(1)	1
C28	-0.5744(7)	-0.0988(4)	-0.3947(4)	0.031(2)	1
C29	-0.4276(8)	-0.0643(4)	-0.3152(4)	0.032(2)	1
C30	-0.3196(8)	0.0353(4)	-0.2602(4)	0.032(2)	1
C31	-0.3755(8)	0.1030(4)	-0.2918(4)	0.032(2)	1
C32	-0.5214(8)	0.0727(4)	-0.3728(4)	0.032(2)	1
C33	-0.1608(8)	0.0675(4)	-0.1793(4)	0.037(2)	1
C34	-0.0256(8)	0.0934(4)	-0.1149(4)	0.034(2)	1
C35	0.1378(7)	0.1256(4)	-0.0368(4)	0.030(2)	1
C36	0.2108(8)	0.0575(4)	-0.0144(4)	0.037(2)	1
C37	0.3642(9)	0.0881(5)	0.0617(5)	0.042(2)	1

C38	0.4518(8)	0.1873(6)	0.1164(4)	0.045(2)	1
C39	0.3848(8)	0.2586(5)	0.0987(4)	0.043(2)	1
C40	0.2300(8)	0.2253(4)	0.0213(4)	0.035(2)	1
F11	-0.3788(4)	-0.1321(2)	-0.2858(2)	0.044(1)	1

U_{eq} is defined as one third of the orthogonalized U_{ij} tensor

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ge1	0.035(1)	0.038(1)	0.037(1)	0.019(1)	0.007(1)	0.017(1)
F1	0.065(3)	0.058(2)	0.055(2)	0.035(2)	0.016(2)	0.032(2)
F2	0.070(3)	0.049(2)	0.067(3)	0.028(2)	0.010(2)	0.013(2)
F3	0.061(3)	0.072(3)	0.054(2)	0.027(2)	-0.005(2)	0.017(2)
F4	0.054(3)	0.067(2)	0.055(2)	0.037(2)	0.008(2)	0.032(2)
F5	0.055(2)	0.047(2)	0.048(2)	0.023(2)	0.016(2)	0.023(2)
F6	0.042(2)	0.041(2)	0.043(2)	0.017(2)	-0.001(2)	0.011(2)
F7	0.052(2)	0.063(2)	0.044(2)	0.033(2)	0.020(2)	0.037(2)
F8	0.050(2)	0.044(2)	0.053(2)	0.029(2)	0.019(2)	0.022(2)
F9	0.048(2)	0.039(2)	0.052(2)	0.026(2)	0.012(2)	0.015(2)
F10	0.044(2)	0.039(2)	0.048(2)	0.018(2)	0.012(2)	0.013(2)
F12	0.061(3)	0.045(2)	0.072(3)	0.031(2)	0.020(2)	0.020(2)
F13	0.066(3)	0.082(3)	0.086(3)	0.063(2)	0.029(2)	0.044(2)
F14	0.053(3)	0.111(3)	0.054(2)	0.039(2)	-0.002(2)	0.020(2)
F15	0.065(3)	0.047(2)	0.063(2)	0.003(2)	0.008(2)	0.002(2)
F16	0.056(3)	0.042(2)	0.072(2)	0.024(2)	0.016(2)	0.023(2)
C1	0.034(4)	0.036(4)	0.028(4)	0.013(3)	0.010(3)	0.018(3)
C2	0.066(5)	0.067(5)	0.028(4)	0.004(4)	0.008(4)	0.040(4)
C3	0.059(5)	0.074(5)	0.048(5)	-0.004(4)	0.000(4)	0.044(4)
C4	0.040(5)	0.054(4)	0.054(5)	0.011(4)	0.017(4)	0.021(4)
C5	0.063(5)	0.043(4)	0.036(4)	0.011(4)	0.017(4)	0.023(4)
C6	0.053(5)	0.035(4)	0.047(5)	0.021(4)	0.013(4)	0.017(4)
C7	0.040(4)	0.033(4)	0.026(4)	0.011(3)	0.009(3)	0.016(3)
C8	0.043(5)	0.042(4)	0.050(4)	0.029(4)	0.008(4)	0.013(4)
C9	0.051(5)	0.059(5)	0.041(4)	0.027(4)	0.018(3)	0.023(4)
C10	0.032(4)	0.062(5)	0.047(4)	0.039(4)	0.008(3)	0.005(4)
C11	0.053(5)	0.042(4)	0.056(5)	0.027(4)	0.017(4)	0.019(4)
C12	0.037(4)	0.036(4)	0.043(4)	0.020(3)	0.009(3)	0.017(3)
C13	0.046(5)	0.069(5)	0.035(4)	0.038(4)	0.015(4)	0.035(4)
C14	0.047(5)	0.042(5)	0.043(4)	0.023(4)	0.014(4)	0.019(4)
C15	0.030(4)	0.052(5)	0.026(4)	0.014(4)	-0.008(3)	0.005(4)
C16	0.044(5)	0.057(5)	0.036(4)	0.031(4)	0.013(4)	0.032(4)
C17	0.041(4)	0.029(4)	0.037(4)	0.018(4)	0.018(4)	0.020(4)
C18	0.029(4)	0.042(4)	0.030(4)	0.013(4)	0.005(3)	0.011(4)
C19	0.046(5)	0.050(4)	0.036(4)	0.019(4)	0.021(4)	0.023(4)
C20	0.031(5)	0.051(4)	0.039(4)	0.023(4)	0.017(4)	0.024(4)
C21	0.022(4)	0.043(4)	0.038(4)	0.021(4)	0.011(3)	0.011(3)
C22	0.038(4)	0.032(4)	0.044(4)	0.024(4)	0.017(4)	0.011(3)
C23	0.030(4)	0.047(4)	0.026(4)	0.021(3)	0.012(3)	0.018(3)
C24	0.028(4)	0.035(4)	0.036(4)	0.016(3)	0.015(3)	0.014(3)
C25	0.034(4)	0.031(4)	0.040(4)	0.023(4)	0.017(3)	0.017(3)
C26	0.028(4)	0.059(5)	0.032(4)	0.035(4)	0.012(3)	0.027(4)
C27	0.023(4)	0.033(4)	0.031(4)	0.014(3)	0.017(3)	0.013(3)
C28	0.027(4)	0.049(4)	0.033(4)	0.024(3)	0.016(3)	0.029(3)

C29	0.038(4)	0.034(4)	0.041(4)	0.023(4)	0.023(4)	0.021(4)
C30	0.037(4)	0.038(4)	0.032(4)	0.019(4)	0.015(3)	0.017(4)
C31	0.029(4)	0.027(4)	0.035(4)	0.009(3)	0.019(3)	0.002(3)
C32	0.036(4)	0.043(5)	0.031(4)	0.023(4)	0.019(3)	0.022(4)
C33	0.037(5)	0.040(4)	0.035(4)	0.013(3)	0.015(4)	0.017(3)
C34	0.034(4)	0.036(4)	0.039(4)	0.019(3)	0.014(4)	0.013(3)
C35	0.032(4)	0.028(4)	0.034(4)	0.015(4)	0.013(3)	0.015(3)
C36	0.044(5)	0.027(4)	0.040(4)	0.013(4)	0.019(4)	0.006(4)
C37	0.041(5)	0.057(5)	0.054(5)	0.042(5)	0.023(4)	0.024(4)
C38	0.027(4)	0.068(5)	0.034(4)	0.025(4)	-0.003(3)	0.010(4)
C39	0.038(5)	0.031(4)	0.046(4)	0.010(4)	0.007(4)	0.001(4)
C40	0.047(5)	0.032(4)	0.042(4)	0.022(4)	0.015(4)	0.028(4)
F11	0.048(2)	0.044(2)	0.042(2)	0.022(2)	0.005(2)	0.021(2)

The general temperature factor expression:

$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}\,hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Ge1	C7	1.917(5)	Ge1	C1	1.931(5)
Ge1	C28	1.933(5)	Ge1	C23	1.949(5)
F1	C13	1.370(5)	F2	C14	1.348(6)
F3	C15	1.342(6)	F4	C16	1.353(5)
F5	C17	1.333(6)	F6	C22	1.358(6)
F7	C26	1.356(5)	F8	C25	1.343(5)
F9	C32	1.356(5)	F10	C31	1.350(5)
F12	C36	1.347(5)	F13	C37	1.348(6)
F14	C38	1.345(6)	F15	C39	1.335(6)
F16	C40	1.345(5)	C1	C2	1.373(6)
C1	C6	1.379(6)	C2	C3	1.378(7)
C2	H2A	0.9300	C3	C4	1.368(7)
C3	H3A	0.9300	C4	C5	1.390(7)
C4	H4A	0.9300	C5	C6	1.390(7)
C5	H5A	0.9300	C6	H6A	0.9300
C7	C12	1.385(6)	C7	C8	1.411(7)
C8	C9	1.389(7)	C8	H8A	0.9300
C9	C10	1.377(6)	C9	H9A	0.9300
C10	C11	1.361(7)	C10	H10A	0.9300
C11	C12	1.381(7)	C11	H11A	0.9300
C12	H12A	0.9300	C13	C14	1.363(7)
C13	C18	1.383(7)	C14	C15	1.373(7)
C15	C16	1.361(7)	C16	C17	1.366(7)
C17	C18	1.411(7)	C18	C19	1.410(7)
C19	C20	1.195(7)	C20	C21	1.426(7)
C21	C26	1.373(7)	C21	C22	1.406(6)
C22	C23	1.376(7)	C23	C24	1.408(7)
C24	C25	1.402(6)	C24	C27	1.498(7)
C25	C26	1.379(7)	C27	C32	1.392(7)
C27	C28	1.433(6)	C28	C29	1.360(6)
C29	F11	1.358(5)	C29	C30	1.388(7)

C30	C31	1.397(6)	C30	C33	1.435(7)
C31	C32	1.381(7)	C33	C34	1.187(7)
C34	C35	1.434(7)	C35	C40	1.368(7)
C35	C36	1.392(7)	C36	C37	1.368(7)
C37	C38	1.349(7)	C38	C39	1.381(7)
C39	C40	1.382(7)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C7	Ge1	C1	114.0(2)	C7	Ge1	C28	110.6(2)
C1	Ge1	C28	114.1(2)	C7	Ge1	C23	114.1(2)
C1	Ge1	C23	114.4(2)	C28	Ge1	C23	86.7(2)
C2	C1	C6	117.8(5)	C2	C1	Ge1	120.4(4)
C6	C1	Ge1	121.8(4)	C1	C2	C3	121.1(5)
C1	C2	H2A	119.5	C3	C2	H2A	119.5
C4	C3	C2	121.5(6)	C4	C3	H3A	119.2
C2	C3	H3A	119.2	C3	C4	C5	118.2(6)
C3	C4	H4A	120.9	C5	C4	H4A	120.9
C4	C5	C6	119.9(5)	C4	C5	H5A	120.1
C6	C5	H5A	120.1	C1	C6	C5	21.4(5)
C1	C6	H6A	119.3	C5	C6	H6A	119.3
C12	C7	C8	116.9(5)	C12	C7	Ge1	23.0(5)
C8	C7	Ge1	120.1(4)	C9	C8	C7	21.5(5)
C9	C8	H8A	119.2	C7	C8	H8A	119.2
C10	C9	C8	118.7(6)	C10	C9	H9A	120.6
C8	C9	H9A	120.6	C11	C10	C9	21.2(6)
C11	C10	H10A	119.4	C9	C10	H10A	119.4
C10	C11	C12	120.0(6)	C10	C11	H11A	120.0
C12	C11	H11A	120.0	C11	C12	C7	21.7(5)
C11	C12	H12A	119.2	C7	C12	H12A	119.2
C14	C13	F1	116.9(6)	C14	C13	C18	24.2(6)
F1	C13	C18	118.8(5)	F2	C14	C13	22.5(6)
F2	C14	C15	118.9(6)	C13	C14	C15	18.6(6)
F3	C15	C16	119.8(6)	F3	C15	C14	20.0(6)
C16	C15	C14	120.2(6)	F4	C16	C15	21.2(6)
F4	C16	C17	118.4(6)	C15	C16	C17	20.4(5)
F5	C17	C16	120.6(5)	F5	C17	C18	17.6(5)
C16	C17	C18	121.8(5)	C13	C18	C19	23.6(6)
C13	C18	C17	114.7(5)	C19	C18	C17	21.7(6)
C20	C19	C18	177.0(6)	C19	C20	C21	177.5(6)
C26	C21	C22	113.9(5)	C26	C21	C20	21.6(5)
C22	C21	C20	124.5(5)	F6	C22	C23	19.5(5)
F6	C22	C21	116.5(5)	C23	C22	C21	24.0(5)
C22	C23	C24	120.3(5)	C22	C23	Ge1	27.0(4)
C24	C23	Ge1	112.7(4)	C25	C24	C23	16.6(5)
C25	C24	C27	129.3(5)	C23	C24	C27	14.1(5)
F8	C25	C26	116.6(5)	F8	C25	C24	22.7(5)
C26	C25	C24	120.7(5)	F7	C26	C21	18.9(5)
F7	C26	C25	116.5(5)	C21	C26	C25	24.5(5)
C32	C27	C28	116.8(5)	C32	C27	C24	28.9(5)
C28	C27	C24	114.3(5)	C29	C28	C27	118.8(5)

C29	C28	Ge1	129.0(4)	C27	C28	Ge1	12.2(4)
F11	C29	C28	118.3(5)	F11	C29	C30	15.8(5)
C28	C29	C30	125.9(5)	C29	C30	C31	14.1(5)
C29	C30	C33	123.8(5)	C31	C30	C33	22.0(5)
F10	C31	C32	118.4(5)	F10	C31	C30	18.7(5)
C32	C31	C30	122.8(5)	F9	C32	C31	15.2(5)
F9	C32	C27	123.2(5)	C31	C32	C27	21.6(5)
C34	C33	C30	177.4(6)	C33	C34	C35	79.5(6)
C40	C35	C36	115.9(5)	C40	C35	C34	21.9(5)
C36	C35	C34	122.1(5)	F12	C36	C37	18.8(6)
F12	C36	C35	119.0(5)	C37	C36	C35	22.2(5)
F13	C37	C38	119.7(6)	F13	C37	C36	20.4(6)
C38	C37	C36	119.8(6)	F14	C38	C37	20.9(6)
F14	C38	C39	118.4(6)	C37	C38	C39	20.7(6)
F15	C39	C38	120.6(6)	F15	C39	C40	21.3(6)
C38	C39	C40	118.0(5)	F16	C40	C35	19.3(5)
F16	C40	C39	117.4(5)	C35	C40	C39	23.2(5)

Table 5. Torsion Angles(^{o})

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C7	Ge1	C1	C2	-64.0(5)	C28	Ge1	C1	C26	4.6(5)
C23	Ge1	C1	C2	162.2(5)	C7	Ge1	C1	C6	118.5(4)
C28	Ge1	C1	C6	-113.0(5)	C23	Ge1	C1	C6	-15.4(5)
C6	C1	C2	C3	-0.3(9)	Ge1	C1	C2	C3	-177.9(5)
C1	C2	C3	C4	0.7(10)	C2	C3	C4	C5	-1.7(10)
C3	C4	C5	C6	2.4(9)	C2	C1	C6	C5	1.1(8)
Ge1	C1	C6	C5	178.7(4)	C4	C5	C6	C1	-2.1(9)
C1	Ge1	C7	C12	-1.7(5)	C28	Ge1	C7	C12	-132.0(4)
C23	Ge1	C7	C12	132.3(4)	C1	Ge1	C7	C8	177.6(4)
C28	Ge1	C7	C8	47.3(5)	C23	Ge1	C7	C8	-48.5(5)
C12	C7	C8	C9	0.7(8)	Ge1	C7	C8	C9	-178.6(4)
C7	C8	C9	C10	-1.7(8)	C8	C9	C10	C11	1.9(8)
C9	C10	C11	C12	-1.2(9)	C10	C11	C12	C7	0.2(8)
C8	C7	C12	C11	0.0(8)	Ge1	C7	C12	C11	179.3(4)
F1	C13	C14	F2	2.0(9)	C18	C13	C14	F2	179.9(5)
F1	C13	C14	C15	179.2(5)	C18	C13	C14	C15	-2.9(10)
F2	C14	C15	F3	-1.4(8)	C13	C14	C15	F3	-178.7(5)
F2	C14	C15	C16	-179.6(5)	C13	C14	C15	C16	3.1(9)
F3	C15	C16	F4	0.4(9)	C14	C15	C16	F4	178.6(5)
F3	C15	C16	C17	179.1(5)	C14	C15	C16	C17	-2.7(9)
F4	C16	C17	F5	0.0(8)	C15	C16	C17	F5	-178.8(5)
F4	C16	C17	C18	-179.3(5)	C15	C16	C17	C18	2.0(9)
C14	C13	C18	C19	-179.3(6)	F1	C13	C18	C19	-1.4(9)
C14	C13	C18	C17	2.1(9)	F1	C13	C18	C17	180.0(4)
F5	C17	C18	C13	179.1(5)	C16	C17	C18	C13	-1.6(8)
F5	C17	C18	C19	0.5(8)	C16	C17	C18	C19	179.8(6)
C13	C18	C19	C20	-155(13)	C17	C18	C19	C20	23(13)
C18	C19	C20	C21	-2(25)	C19	C20	C21	C26	-24(15)
C19	C20	C21	C22	156(15)	C26	C21	C22	F6	-179.1(5)
C20	C21	C22	F6	1.1(8)	C26	C21	C22	C23	1.4(8)
C20	C21	C22	C23	-178.4(5)	F6	C22	C23	C24	179.8(5)
C21	C22	C23	C24	-0.7(9)	F6	C22	C23	Ge1	-1.1(8)
C21	C22	C23	Ge1	178.4(4)	C7	Ge1	C23	C22	-69.4(5)

C1	Ge1	C23	C22	64.5(6)	C28	Ge1	C23	C22	179.5(5)
C7	Ge1	C23	C24	109.8(4)	C1	Ge1	C23	C24	-116.3(4)
C28	Ge1	C23	C24	-1.3(4)	C22	C23	C24	C25	0.1(8)
Ge1	C23	C24	C25	-179.2(4)	C22	C23	C24	C27	-179.8(5)
Ge1	C23	C24	C27	0.9(6)	C23	C24	C25	F8	-178.6(5)
C27	C24	C25	F8	1.3(8)	C23	C24	C25	C26	-0.2(8)
C27	C24	C25	C26	179.7(5)	C22	C21	C26	F7	-179.7(4)
C20	C21	C26	F7	0.2(8)	C22	C21	C26	C25	-1.5(9)
C20	C21	C26	C25	178.3(5)	F8	C25	C26	F7	-2.3(7)
C24	C25	C26	F7	179.2(4)	F8	C25	C26	C21	179.5(5)
C24	C25	C26	C21	1.0(9)	C25	C24	C27	C32	-0.1(9)
C23	C24	C27	C32	179.8(5)	C25	C24	C27	C28	-179.7(5)
C23	C24	C27	C28	0.2(7)	C32	C27	C28	C29	0.1(7)
C24	C27	C28	C29	179.7(5)	C32	C27	C28	Ge1	179.1(4)
C24	C27	C28	Ge1	-1.2(5)	C7	Ge1	C28	C29	65.8(5)
C1	Ge1	C28	C29	-64.5(5)	C23	Ge1	C28	C29	-179.7(5)
C7	Ge1	C28	C27	-113.2(4)	C1	Ge1	C28	C27	116.6(4)
C23	Ge1	C28	C27	1.4(4)	C27	C28	C29	F11	-179.8(4)
Ge1	C28	C29	F11	1.3(8)	C27	C28	C29	C30	-0.3(9)
Ge1	C28	C29	C30	-179.1(4)	F11	C29	C30	C31	-179.1(4)
C28	C29	C30	C31	1.3(9)	F11	C29	C30	C33	2.6(8)
C28	C29	C30	C33	-177.0(5)	C29	C30	C31	F10	-178.9(5)
C33	C30	C31	F10	-0.5(8)	C29	C30	C31	C32	-2.3(8)
C33	C30	C31	C32	176.1(5)	F10	C31	C32	F9	-1.9(7)
C30	C31	C32	F9	-178.5(4)	F10	C31	C32	C27	178.9(5)
C30	C31	C32	C27	2.3(9)	C28	C27	C32	F9	179.8(4)
C24	C27	C32	F9	0.2(9)	C28	C27	C32	C31	-1.1(8)
C24	C27	C32	C31	179.3(5)	C29	C30	C33	C34	88(13)
C31	C30	C33	C34	-91(13)	C30	C33	C34	C35	109(82)
C33	C34	C35	C40	-8(87)	C33	C34	C35	C36	170(100)
C40	C35	C36	F12	-179.7(5)	C34	C35	C36	F12	1.3(8)
C40	C35	C36	C37	0.7(8)	C34	C35	C36	C37	-178.3(5)
F12	C36	C37	F13	0.8(8)	C35	C36	C37	F13	-179.6(5)
F12	C36	C37	C38	179.0(5)	C35	C36	C37	C38	-1.4(9)
F13	C37	C38	F14	-0.7(9)	C36	C37	C38	F14	-178.8(5)
F13	C37	C38	C39	-179.6(5)	C36	C37	C38	C39	2.3(10)
F14	C38	C39	F15	1.9(9)	C37	C38	C39	F15	-179.2(6)
F14	C38	C39	C40	178.7(5)	C37	C38	C39	C40	-2.4(9)
C36	C35	C40	F16	179.3(4)	C34	C35	C40	F16	-1.6(8)
C36	C35	C40	C39	-0.9(9)	C34	C35	C40	C39	178.2(5)
F15	C39	C40	F16	-1.7(8)	C38	C39	C40	F16	-178.5(5)
F15	C39	C40	C35	178.5(5)	C38	C39	C40	C35	1.7(9)