Electronic Supplementary Material (ESI) for ChemComm.

SUPPLEMENTARY INFORMATION

Polar alicyclic rings: Synthesis and structure of all *syn*-1,2,3,4-tetrafluorocyclopentane

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Table of content

1.General Experimental Notes

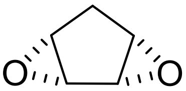
All chemicals were purchased from Acros, Sigma Aldrich, Alfa Aeser, Fisher Scientific, Fluorochem or Merck. Triphenylphosphite was purified by separation between diethyl ether and 5% aq. NaOH, then the organic layer was washed with water and sat. aq. NaCl, dried over Na_2SO_4 and the solvent removed under reduced pressure. All non-aqueous reactions were carried out in oven-dried glassware under an inert argon atmosphere. Anhydrous CH_2Cl_2 was obtained from a MBraun MB SPS-800 solvent purification system dispensed under an atmosphere of argon gas. Thin layer chromatography was carried out on aluminium backed Merck TLC silica gel 60 F_{254} plates. These plates were visualised using UV light at a wavelength of 254 nm, potassium permanganate or phosphomolybdic acid.

All NMR spectra were recorded using either a Bruker Avance II 400, Bruker Avance 300 or 500 spectrometers in deuterated solvents.^{1}H NMR spectra were recorded at either 300, 400 or 500 MHz.^{13}C NMR spectra were recorded using the UDEFT pulse sequence and broadband proton decoupling at either 75, 100 or 126 MHz.^{19}F NMR spectra were recorded at 282, 376 or 470 MHz. All chemical shifts, δ, are stated in units of parts per million (ppm), relative to a standard reference point, for^{1}H NMR and^{13}C NMR the reference point is TMS, where δ_H and δ_C is 0.00 ppm. For^{19}F NMR the reference point is CCl_3F, where δ_F is 0.00 ppm.

Melting points were determined using a Reichart Hot Stage Microscope and are uncorrected. High and low resolution mass spectra were obtained by atmospheric pressure chemical ionisation(APCI), electrospray ionization(ESI) and electron ionization(EI). ESI-MS spectra were recorded on a Waters Micromass LCT spectrometer in positive mode or negative mode. EI-MS spectra were recorded on a Waters Micromass GCT spectrometer. Values are reported as a ratio of mass to charge(m/z).

2. Procedures and analytical data

***cis 1,2,3,4-diepoxycyclopentane* (8)** [1,2]



Ozone was bubbled through a solution of triphenylphosphite (14.8 g, 12.5 mL, 47.7 mmol) in dry methylene chloride (150 mL) at -78 °C. When the blue colour of excess of ozone was observed, oxygen was bubbled through the solution until the blue colour has been completely disappeared at maintain temperature. The solution was warmed to -30 °C, stirred for 10 min, then cooled to -78 °C again, and cyclopentadiene (3 g, 3.82 mL, 45.5 mmol) was added and the solution was stirred at maintain temperature for 1h. Thereafter a solution of $RuCl_2(PPh_3)_3$ (0.5 g, 0.75 mmol) in methylene chloride (5 mL) was quickly added. The reaction mixture was stirred at -78 °C for 1h, at -40 °C for 0.5h and finally at -25 °C for 1h. Solvent was carefully removed under reduced pressure, and the dark brown residue was subjected to column chromatography (petroleum ether/diethyl ether 1:1) to give the product (1.7 g, 38%) as yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$)** δ_H 1.65 (1 H, dt, J = 16.3, 3.0 Hz, CH_2), 2.12 (1 H, d, J = 16.3 Hz, CH_2), 3.53 (2 H, dd, J = 1.9, 1.2 Hz, 2CHO), 3.74 (2 H, ddd, J = 4.2, 1.9, 1.1 Hz, 2CHO); **^{13}C NMR (100 MHz, $CDCl_3$)** δ_c 65.26 (CHO) 52.16 (CHO) 28.35 (CH_2); **HRMS** m/z [Found: $[M+Na]^+$ 121.0259. $C_5H_6Na^+$ requires m/z 121.0265]. These data are in good agreement with the literature data[2].

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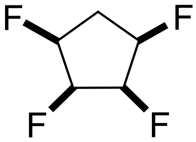
***(1R,2R,3S,4S)-1,2,3,4-Tetrafluorocyclopentane* (5)**

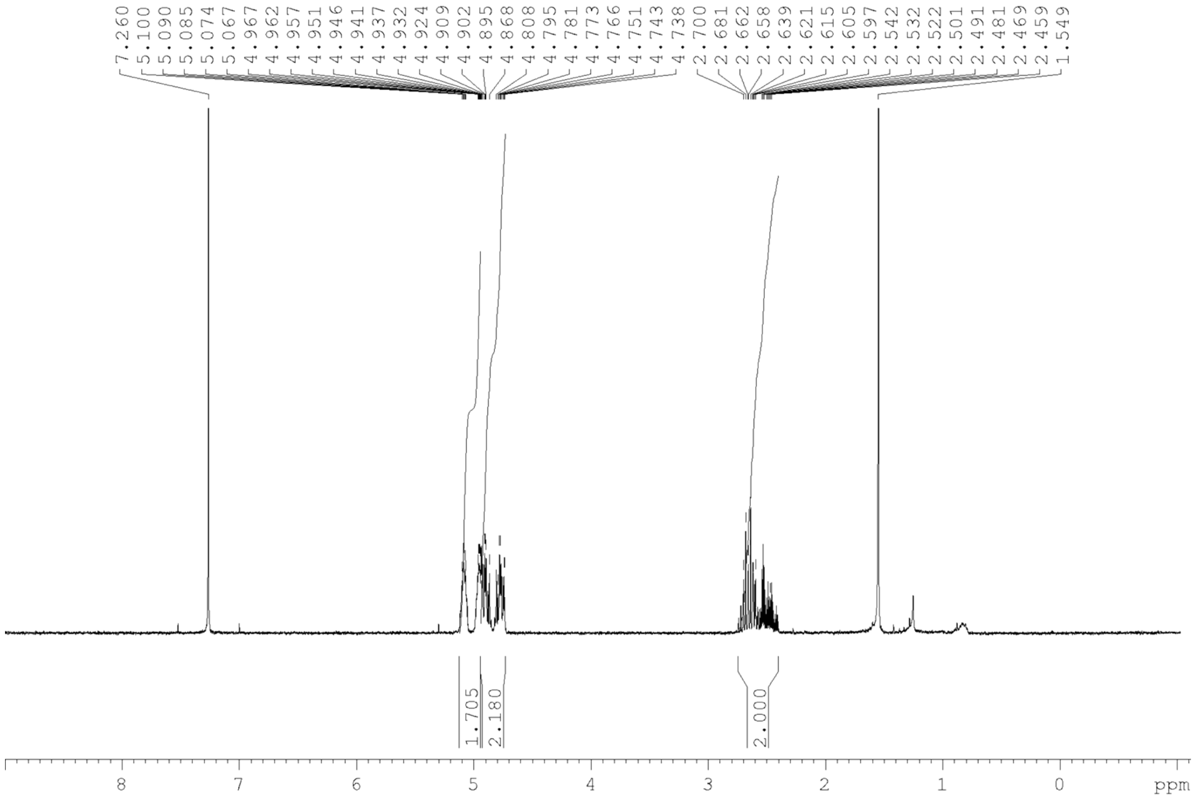


A solution of diepoxide (0.9 g, 9.1 mmol) in $Et_3N\cdot 3HF$ (12 mL, 68.3 mmol) in a Teflon flask was stirred at 120 ℃ for 24h. The reaction mixture was poured into water (10 mL) and extracted with diethyl ether (20 mL x 3). The combined organic layers were dried over magnesium sulphate, filtered and the solvent was removed under reduced pressure to give a mixture of **9** and **10** (2.1 g) in 2:1 ratio. The mixture was dissolved in anhydrous methylene chloride (25 mL), trifluomethanesulfonic anhydride (15.4 mL, 91.3 mmol) and pyridine (5 mL, 60.9 mmol) were added subsequently under argon atmosphere at 0 ºC. After 24h stirring at room temperature, the mixture was extracted with petroleum ether and diethyl ether (150 mL x 3) then filtered. The solvent was removed under reduced pressure to give a mixture of triflates **11** and **12** (2.2 g), which was used for reaction without further purification. The triflates and $Et_3N\cdot 3HF$ (8.9 mL, 54 mmol) were placed in a Teflon flask and stirred at 100 ºC under argon atmosphere for 48h. The reaction mixture was poured into water (20 mL) and extracted with ether (20 mL x 3). The combined organic extracts were dried over sodium sulphate, filtered, and concentrated under reduced pressure (500 mbar) at room temperature. The residue was purified by silica gel chromatography (petroleum ether/ diethyl ether 3:2) to give the desired tetrafluorocyclopentane (113 mg, 8.7%) as a white solid. **Mp =** **39-40 ºC;** **^{1}H NMR (400 Hz, CDCl$_3$)** δ_H 2.40-2.74 (2 H, m, CH_2), 4.76-4.93 (2 H, dm, CHF), 4.92-5.12 (2 H, dm, CHF); **^{1}H {^{19}F}NMR (400 Hz, CDCl$_3$)** δ_H 2.51 (1 H, dt, J = 16.3, 4.3 Hz, CH_2), 2.66 (1 H, dt, J = 16.3, 7.4 Hz, CH_2), 4.84 (2 H, dd, J = 3.2, 1.8 Hz, CHO), 5.02 (2 H, m, CHO); **^{13}C NMR (100 Hz, CDCl$_3$)** δ_c 35.9 (t, J = 23.1 Hz, $\underline{C}H_2$), 87.8 (dm, J = 195.4 Hz, $\underline{C}$HF), 88.3 (dm, J = 203.4 Hz, $\underline{C}$HF), **^{19}F {^{1}H}NMR (376 Hz, CDCl$_3$)** δ_F **-**196.2 (d, J = 3.2 Hz, CH$\underline{F}$), -219.4 (d, J = 3.2 Hz, CH$\underline{F}$); **^{19}F NMR (376 Hz, CDCl$_3$)** δ_F **-**196.2 (m, CH$\underline{F}$), -219.4 (m, CH$\underline{F}$); **HRMS** *m/z* [Found: $[M+Na]^+$ 165.0297. $C_5H_6F_4Na^+$ requires m/z 165.0303].

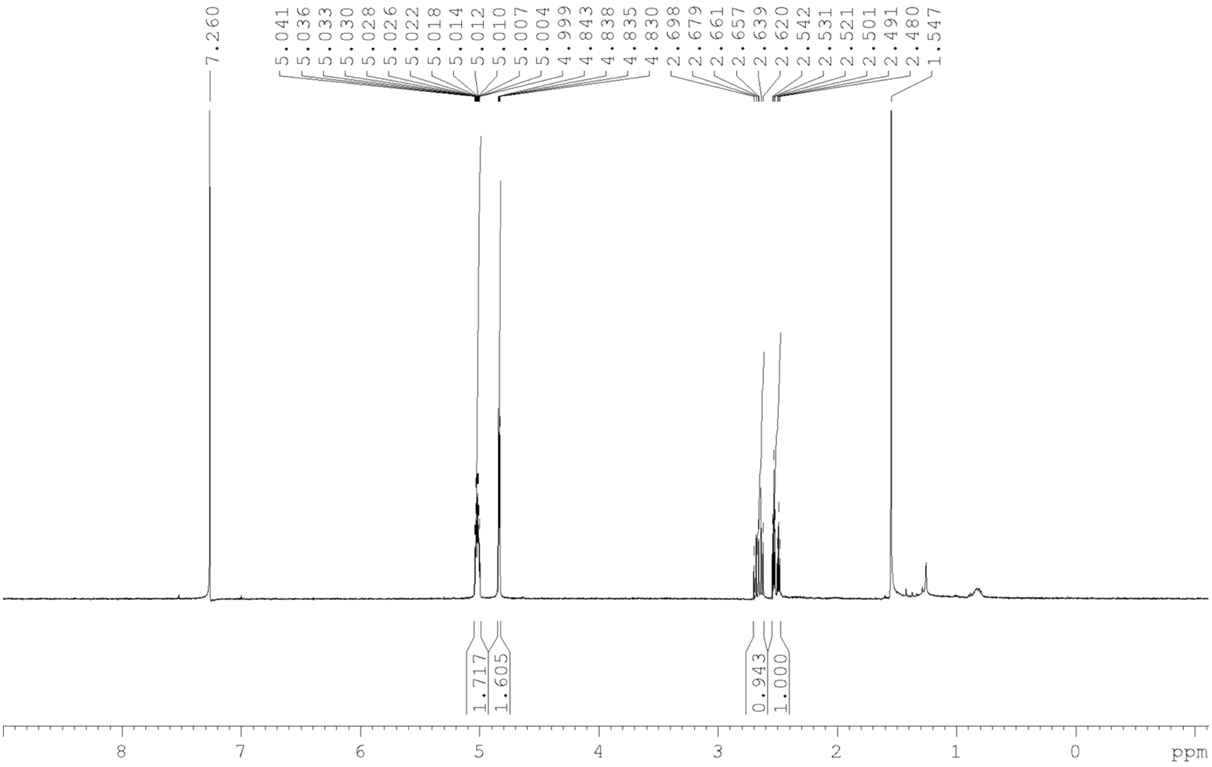
3. NMR Spectra

(1R,2R,3S,4S)-1,2,3,4-Tetrafluorocyclopentane

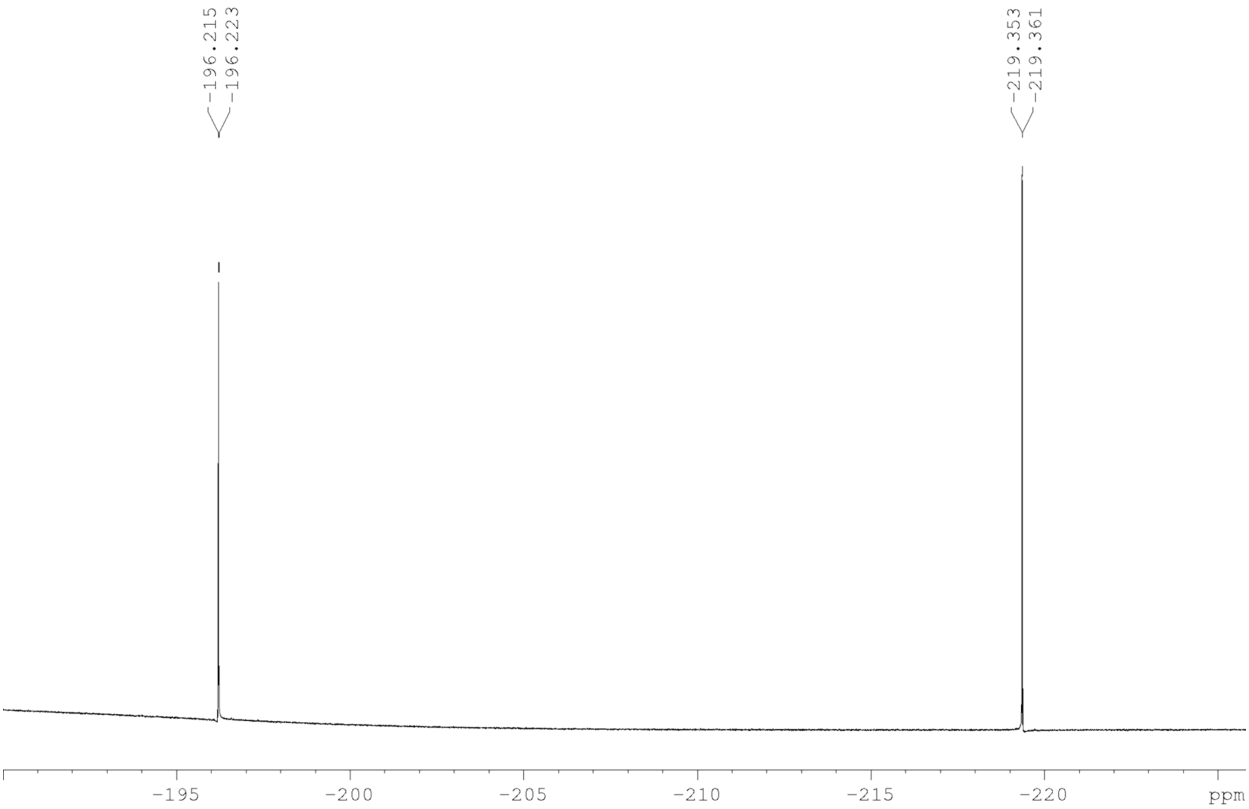
^{1}H NMR



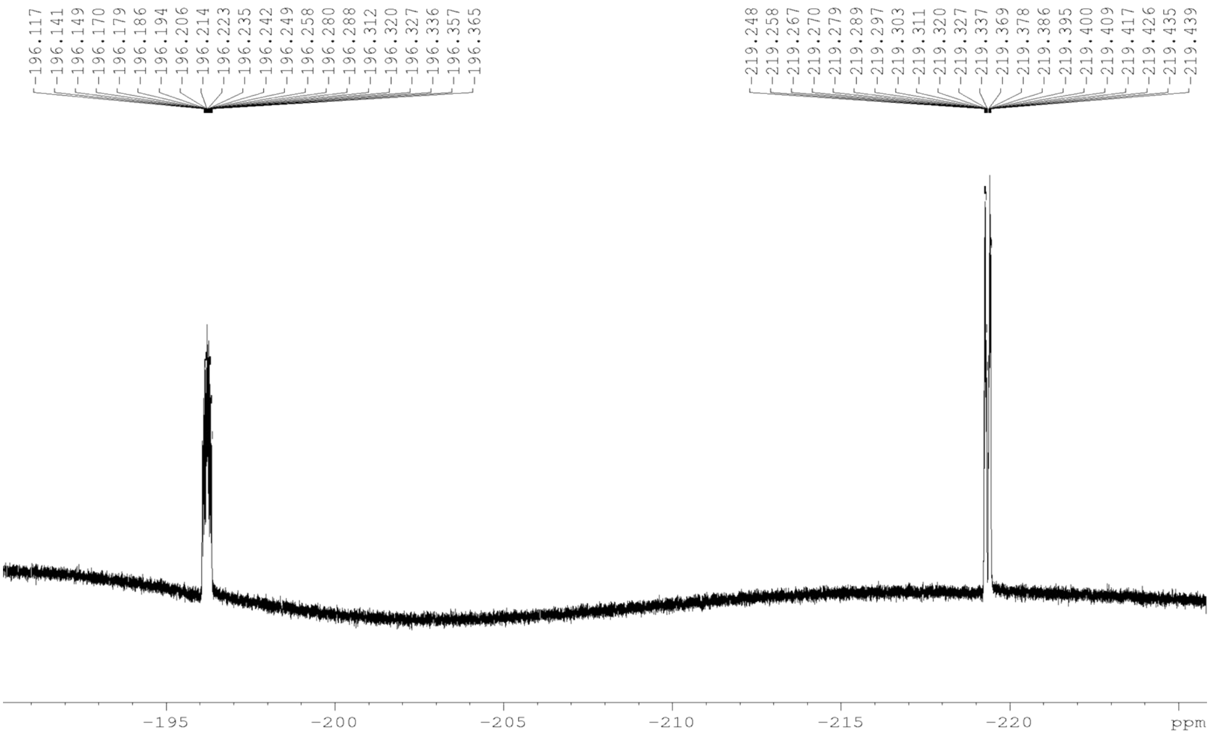
^{1}H {^{19}F}NMR



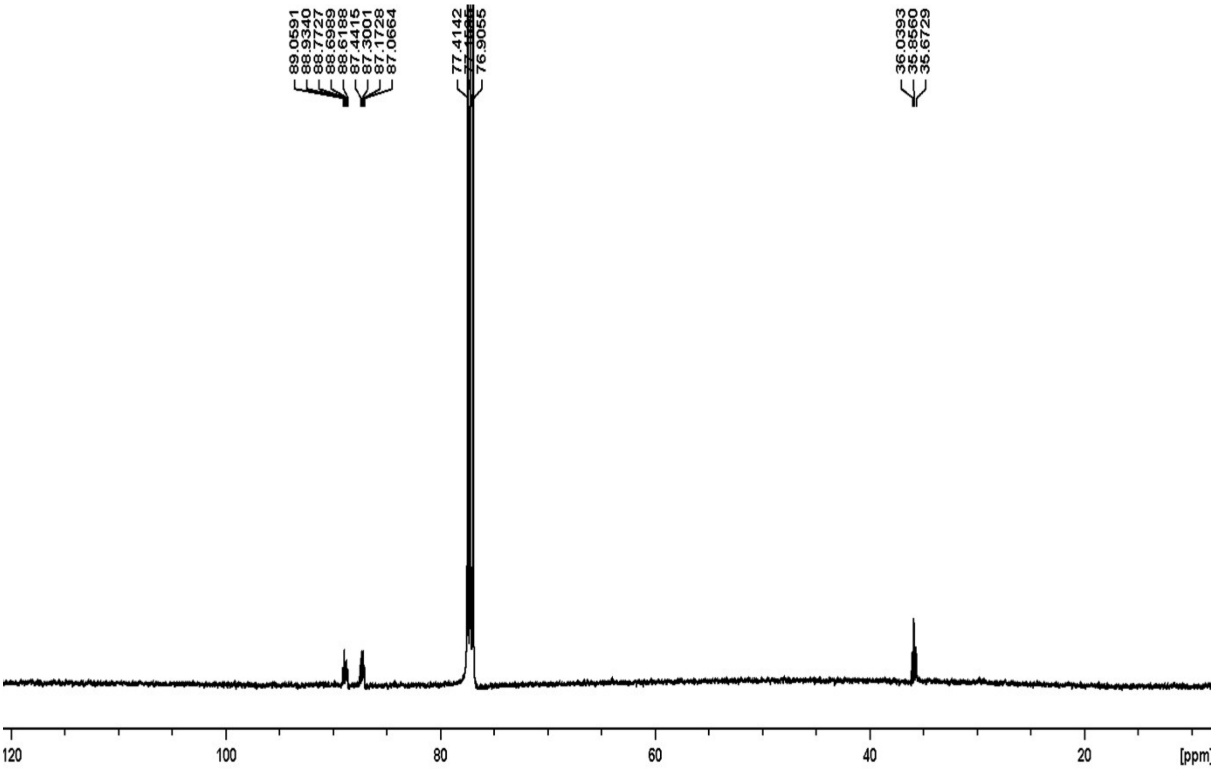
^{19}F {^{1}H}NMR



^{19}F NMR



^{13}C NMR


89.0591
88.9340
88.7727
88.6989
88.6188
87.4415
87.3001
87.1728
87.0664
77.4142
76.9055
36.0393
35.8560
35.6729
120
100
80
60
40
20
[ppm]

4. Computational Details

Full geometry optimisations were performed in the gas phase starting from a single monomer taken from the X-ray structure. Using 6-311+G(d,p) basis, a variety of DFT (B3LYP, [1,2] B3LYP-D3,[3] BLYP-D2,[4,2,5]M06-2X,[6] M11[7]) and ab initio levels (MP2) were trialled, all of which afforded essentially the same minimum, **5a**. Additional optimisations were carried out at the B3LYP-D3/6-311+G(d,p) level for an isolated stacked dimer taken from the X-ray structure, as well as for a monomer using the CPCM variant of a polarisable continuum model,[8] employing parameters of solvents with increasing polarity (dichloromethane, acetonitrile and water). In none of these calculations the conformation observed in the solid was maintained, all optimised to minima resembling **5a**. The X-ray conformation was then kept frozen by fixing the five C-C-C-C dihedral angles to the values observed in the solid, relaxing all other geometrical parameters. Additional conformers were sought at the B3LYP-D3/6-311+G(d,p) level by unconstrained optimisations in the gas phase starting from envelope conformations of **5** with different placements of the F atoms (maintaining all-cis stereochemistry). All of these optimisations converged to the same minimum, **5a**. For two of the structures the starting conformation could be maintained by imposing C_s symmetry, denoted **5b** and **5b'**. Computed harmonic vibrational frequencies at the B3LYP-D3 level showed that **5a** is a true minimum and **5b** and **5b'** are transition states, and were used to evaluate thermodynamic corrections affording computed enthalpies of activation. A plot of the electrostatic potential of **5a** is shown in Figure S1, showing the "negative" and "positive faces".

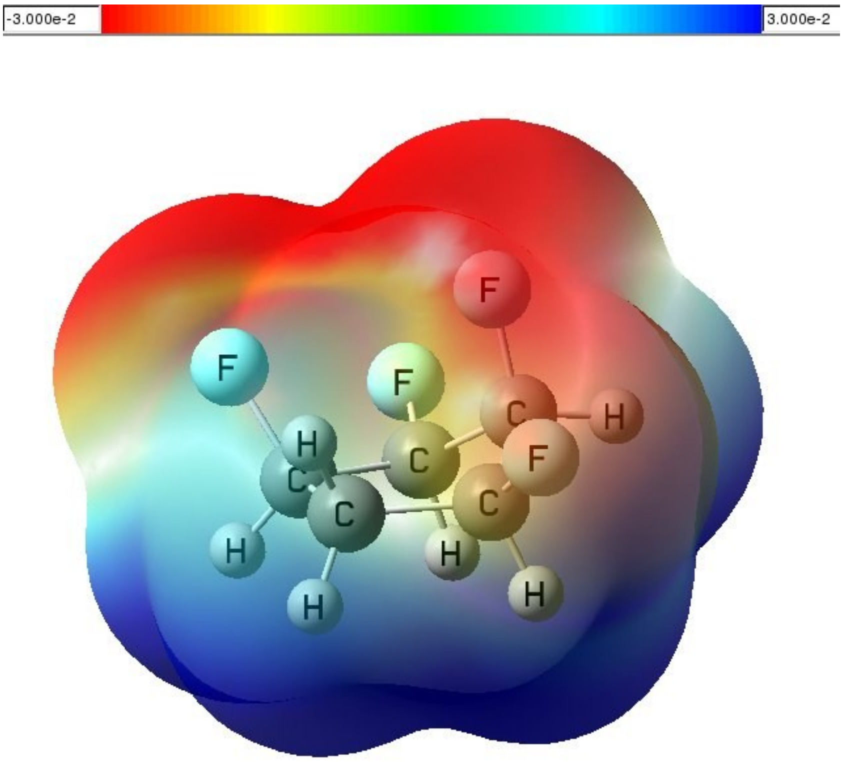
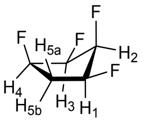


Figure S1: Electrostatic potential for **5a** at the B3LYP-D3/6-311+G(d,p) level, plotted on a colour scale from -0.003 a.u. (red) to +0.003 a.u. (blue) and mapped onto an isodensity surface ($\rho = 4{\cdot}10^{-4}$ a.u.).

Indirect spin-spin coupling constants were computed at the BHandH/6-311+G(2d,p) level[9] for B3LYP-D3/6-311+G(d,p) optimised structures. The averaged results involving the protons of the CH_2 group are given in the main paper, all individual couplings are collected in Table S1. These calculations were performed using the Gaussian 09 program.[10]

Table S1: Selected computed J(H,H) coupling constants (in Hz), BHandH/6-311+G(2d,p) level (B3LYP-D3/6-311+G(d,p) geometries).

 **5**	H_{5a}-H_{5b}	H_{5a}-H_4	H_{5a}-H_1	H_{5a}-$H_{1,4}$[a]	H_{5b}-H_4	H_{5b}-H_1	H_{5b}-$H_{1,4}$[a]	**MAD**[b]
Expt[c] **5**	*16.3*			*4.3*			*7.4*	*n.a.*
Calc. **5a**	-15.1	1.3	7.7	4.5	6.7	8.5	7.6	**0.5**
Calc. **5**(fix)[d]	-15.8	0.1	3.4	1.8	5.5	9.6	7.6	**1.1**
Calc. **5b**	-10.9	10.8	10.8	10.8	5.6	5.6	5.6	**5.5**

[a]Mean value. [b]**Mean absolute deviation** from experiment [absolute values used for $J(H_{5a},H_{5b})$]. [c] Experimental data from section 2 above (^{19}F-decoupled, $CDCl_3$). [d]Partially optimised structure with C-C-C-C dihedrals fixed to their values observed in the solid.

Optimised Cartesian Coordinates

B3LYP-D3/6-311+G(d,p) level, xyz format in Å

```
15
5a
F   1.415005   1.213656   0.970887
F   2.063152  -1.046622  -0.348213
F  -0.295545  -1.177378   1.169531
F  -2.560790  -0.067116   0.152642
C   0.853229   1.073208  -0.295043
H   1.422567   1.725486  -0.958357
C   0.914199  -0.399708  -0.740455
H   0.871681  -0.424893  -1.837039
C  -0.367553  -1.033264  -0.207926
H  -0.596283  -2.010750  -0.633965
C  -1.377841   0.063869  -0.559125
H  -1.637092  -0.016675  -1.619433
C  -0.656355   1.386599  -0.247338
H  -0.902791   1.692431   0.770754
H  -0.948559   2.187309  -0.926253
```

15

5a(fix)

F	1.166426	1.146803	1.134825
F	2.110573	-0.905218	-0.266147
F	-0.248948	-1.348593	0.978402
F	-2.439705	-0.135112	0.314478
C	0.821688	1.136271	-0.206488
H	1.416124	1.851754	-0.776149
C	0.978597	-0.293100	-0.757080
H	1.042643	-0.247928	-1.851126
C	-0.314035	-1.034536	-0.362808
H	-0.489224	-1.915349	-0.982450
C	-1.392410	0.046262	-0.570090
H	-1.768183	-0.011581	-1.595466
C	-0.686736	1.403701	-0.363069
H	-1.073889	1.896008	0.529453
H	-0.865201	2.054599	-1.221079

15

5b

F	1.504100	0.448983	1.246290
F	-0.859574	0.278175	2.383672
C	0.737821	-0.601650	0.782596
H	1.149736	-1.517382	1.208943
C	-0.741470	-0.370799	1.167907
H	-1.236859	-1.342592	1.275169
C	-1.335058	0.407055	0.000000
H	-2.423978	0.428350	0.000000
C	-0.741470	-0.370799	-1.167907
H	-1.236859	-1.342592	-1.275169
C	0.737821	-0.601650	-0.782596
H	1.149736	-1.517382	-1.208943
F	-0.859574	0.278175	-2.383672
F	1.504100	0.448983	-1.246290
H	-0.949096	1.429818	0.000000

15

5b' (alternative conformer)

C	0.737856	-0.601604	0.782622
C	-0.741452	-0.371117	1.168037
C	-1.335399	0.406231	0.000000
H	-2.424305	0.427384	0.000000
C	-0.741452	-0.371117	-1.168037
C	0.737856	-0.601604	-0.782622
H	-0.949793	1.429215	0.000000
H	-1.236627	-1.342783	-1.276365
H	-1.236627	-1.342783	1.276365
H	1.149762	-1.517392	1.208831
H	1.149762	-1.517392	-1.208831
F	-0.859351	0.278779	2.383744
F	-0.859351	0.278779	-2.383744
F	1.503983	0.448945	1.246270
F	1.503983	0.448945	-1.246270

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5. X-Ray Data Collection for Structure 5

Data Collection

A colourless prism crystal of $C_5H_6F_4$ having approximate dimensions of 0.150 x 0.030 x 0.030 mm was mounted in a loop. All measurements were made on a Rigaku XtaLAB P200 diffractometer using multi-layer mirror monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 1504 carefully centered reflections in the range $4.01 < 2\theta < 55.15^{o}$ corresponded to a primitive monoclinic cell with dimensions:

$a = 10.1150(19)$ Å
$b = 4.5875(11)$ Å $\beta = 90.026(18)^{o}$
$c = 11.876(3)$ Å
$V = 551.1(2)$ Å^3

For Z = 4 and F.W. = 142.10, the calculated density is 1.713 g/cm^3. The reflection conditions of:

h0l: l = 2n
0k0: k = 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -180 $\pm$ 1^{o}C using the ω-2θ scan technique to a maximum 2θ value of 56.5^{o}.

Data Reduction

Of the 3171 reflections were collected, where 1138 were unique (R_{int} = 0.0922). Data were collected and processed using CrysAlisPro (Rigaku Oxford Diffraction). [1] No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.937 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.606 to 0.994. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 987 observed reflections and 83 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0605$$

$$wR2 = [\, \Sigma \, (w \, (Fo^2 - Fc^2)^2 \,) / \, \Sigma \, w(Fo^2)^2]^{1/2} = 0.1610$$

The goodness of fit[4] was 1.05. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.37 and -0.39 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL Version 2014/7[10].

References

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(3) Least Squares function minimized: (SHELXL Version 2014/7)

$$\Sigma w(F_o^2-F_c^2)^2 \quad \text{where w = Least Squares weights.}$$

(4) Goodness of fit is defined as:

$$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where: N_O = number of observations
N_V = number of variables

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_5H_6F_4$
Formula Weight	142.10
Crystal Color, Habit	colourless, prism
Crystal Dimensions	0.150 X 0.030 X 0.030 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	1504 (4.0 - 55.2^{o})
Lattice Parameters	a = 10.1150(19) Å b = 4.5875(11) Å c = 11.876(3) Å β = 90.026(18) ^{o} V = 551.1(2) Å^3
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.713 g/cm^3
F_{000}	288.00
μ(MoKα)	1.937 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB P200
Radiation	MoKα (λ = 0.71075 Å) multi-layer mirror monochromated
Voltage, Current	45kV, 66mA
Temperature	-180.0^{o}C
Detector Aperture	83.8 x 70.0 mm
Pixel Size	0.172 mm
$2\theta_{max}$	50.4^{o}
No. of Reflections Measured	Total: 2971 Unique: 987 (R_{int} = 0.0922)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.606 - 0.994)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.0695 \cdot P)^2$ $+ 0.0000 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	50.4°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	987
No. Variables	83
Reflection/Parameter Ratio	11.89
Residuals: R1 ($I>2.00\sigma(I)$)	0.0605
Residuals: R (All reflections)	0.0638
Residuals: wR2 (All reflections)	0.1610
Goodness of Fit Indicator	1.049
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.37 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.39 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
F1	0.76155(18)	0.0440(4)	0.9837(2)	3.37(5)
F2	0.98770(16)	0.2752(4)	0.91316(18)	2.93(4)
F3	0.82840(18)	0.0525(4)	0.75088(17)	3.10(5)
F4	0.60053(19)	0.2811(5)	0.69819(19)	4.12(5)
C1	0.7583(3)	0.3459(7)	0.9627(3)	2.40(6)
C2	0.8719(3)	0.4256(6)	0.8850(3)	2.37(6)
C3	0.8235(3)	0.3555(7)	0.7663(3)	2.37(6)
C4	0.6776(3)	0.4521(7)	0.7703(3)	2.47(6)
C5	0.6353(3)	0.4226(7)	0.8926(3)	2.80(6)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.76154	0.46053	1.03424	2.875
H2	0.88880	0.63984	0.89068	2.848
H3	0.87471	0.46253	0.70735	2.838
H4	0.67106	0.66109	0.74695	2.959
H5A	0.56796	0.26724	0.90010	3.360
H5B	0.59643	0.60801	0.91952	3.360

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F1	0.0369(11)	0.0331(14)	0.0581(14)	0.0033(8)	0.0131(10)	0.0049(9)
F2	0.0176(9)	0.0413(10)	0.0524(12)	0.0086(6)	-0.0013(9)	-0.0021(10)
F3	0.0388(10)	0.0303(12)	0.0488(12)	0.0032(7)	0.0002(10)	-0.0045(9)
F4	0.0256(10)	0.0770(15)	0.0538(14)	-0.0153(8)	-0.0002(11)	-0.0139(11)
C1	0.0226(15)	0.0264(18)	0.0420(19)	0.0040(12)	0.0060(13)	0.0003(14)
C2	0.0131(12)	0.0289(16)	0.0482(18)	0.0044(11)	0.0014(12)	0.0017(15)
C3	0.0215(13)	0.0234(16)	0.0449(19)	-0.0000(12)	0.0029(13)	-0.0033(14)
C4	0.0183(13)	0.0291(19)	0.0464(19)	-0.0036(11)	-0.0036(13)	0.0011(14)
C5	0.0176(14)	0.0407(19)	0.0481(19)	0.0020(12)	0.0021(13)	-0.0074(16)

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 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
F1	C1	1.408(4)	F2	C2	1.400(3)
F3	C3	1.403(4)	F4	C4	1.399(4)
C1	C2	1.518(4)	C1	C5	1.537(4)
C2	C3	1.526(5)	C3	C4	1.541(4)
C4	C5	1.521(5)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	1.000	C2	H2	1.000
C3	H3	1.000	C4	H4	1.000
C5	H5A	0.990	C5	H5B	0.990

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	C2	109.1(2)	F1	C1	C5	109.9(2)
C2	C1	C5	103.2(3)	F2	C2	C1	111.7(3)
F2	C2	C3	112.7(2)	C1	C2	C3	105.6(2)
F3	C3	C2	108.5(3)	F3	C3	C4	108.9(2)
C2	C3	C4	102.6(3)	F4	C4	C3	110.7(2)
F4	C4	C5	112.2(2)	C3	C4	C5	105.9(3)
C1	C5	C4	108.0(2)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
F1	C1	H1	111.5	C2	C1	H1	111.5
C5	C1	H1	111.5	F2	C2	H2	108.9
C1	C2	H2	109.0	C3	C2	H2	109.0
F3	C3	H3	112.1	C2	C3	H3	112.1
C4	C3	H3	112.1	F4	C4	H4	109.3
C3	C4	H4	109.3	C5	C4	H4	109.3
C1	C5	H5A	110.1	C1	C5	H5B	110.1
C4	C5	H5A	110.1	C4	C5	H5B	110.1
H5A	C5	H5B	108.4				

Table 8. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
F1	C1	C2	F2	-40.6(3)	F1	C1	C2	C3	82.2(3)
F1	C1	C5	C4	-99.3(3)	C2	C1	C5	C4	16.9(3)
C5	C1	C2	F2	-157.3(2)	C5	C1	C2	C3	-34.6(3)
F2	C2	C3	F3	45.9(3)	F2	C2	C3	C4	161.0(2)
C1	C2	C3	F3	-76.2(3)	C1	C2	C3	C4	38.9(3)
F3	C3	C4	F4	-34.5(3)	F3	C3	C4	C5	87.4(3)
C2	C3	C4	F4	-149.3(2)	C2	C3	C4	C5	-27.5(3)
F4	C4	C5	C1	127.6(2)	C3	C4	C5	C1	6.7(3)

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	F2	2.657(3)	F1	F3	2.847(3)
F1	C3	3.017(4)	F1	C4	3.263(4)
F2	F3	2.711(3)	F3	F4	2.608(3)
F3	C1	2.940(4)	F3	C5	3.088(4)
F4	C1	3.535(4)	F4	C2	3.590(4)

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H2	3.217	F1	H5A	2.422
F1	H5B	3.172	F2	H1	2.833
F2	H3	2.831	F3	H2	3.223
F3	H4	3.214	F3	H5A	3.325
F4	H3	2.897	F4	H5A	2.421
F4	H5B	3.026	C1	H3	3.297
C1	H4	3.071	C2	H4	2.824
C2	H5A	3.164	C2	H5B	2.938
C3	H1	3.279	C3	H5A	3.061
C3	H5B	3.151	C4	H1	3.248
C4	H2	2.710	C5	H2	2.751
C5	H3	3.278	H1	H2	2.290
H1	H5A	2.675	H1	H5B	2.259
H2	H3	2.329	H2	H4	2.788
H2	H5B	2.981	H3	H4	2.301
H4	H5A	2.768	H4	H5B	2.198

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	F2^{1}	3.174(3)	F1	F4^{2}	3.129(3)
F1	C1^{3}	3.212(4)	F1	C2^{3}	3.266(4)
F1	C3^{2}	3.445(4)	F1	C4^{2}	3.508(4)
F1	C5^{3}	3.305(4)	F2	F1^{1}	3.174(3)
F2	F2^{1}	3.270(3)	F2	F2^{4}	2.928(3)
F2	F3^{5}	2.979(3)	F2	C1^{4}	3.434(4)
F2	C2^{4}	3.106(4)	F2	C3^{6}	3.450(4)
F3	F2^{6}	2.979(3)	F3	C1^{7}	3.526(4)
F3	C2^{3}	3.316(4)	F3	C2^{6}	3.484(3)
F3	C3^{3}	3.203(4)	F3	C4^{3}	3.156(4)
F4	F1^{7}	3.129(3)	F4	F4^{8}	3.304(3)
F4	F4^{9}	3.304(3)	F4	C1^{7}	3.273(4)
F4	C4^{8}	3.215(4)	F4	C5^{8}	3.091(4)
C1	F1^{10}	3.212(4)	C1	F2^{4}	3.434(4)
C1	F3^{2}	3.526(4)	C1	F4^{2}	3.273(4)
C2	F1^{10}	3.266(4)	C2	F2^{4}	3.106(4)
C2	F3^{10}	3.316(4)	C2	F3^{5}	3.484(3)
C3	F1^{7}	3.445(4)	C3	F2^{5}	3.450(4)
C3	F3^{10}	3.203(4)	C4	F1^{7}	3.508(4)
C4	F3^{10}	3.156(4)	C4	F4^{9}	3.215(4)
C5	F1^{10}	3.305(4)	C5	F4^{9}	3.091(4)

Symmetry Operators:

(1) -X+2,-Y,-Z+2 (2) X,-Y+1,Z+1
(3) X,Y-1,Z (4) -X+2,-Y+1,-Z+2
(5) -X+2,Y+1/2,-Z+1/2+1 (6) -X+2,Y+1/2-1,-Z+1/2+1
(7) X,-Y+1,Z (8) -X+1,Y+1/2-1,-Z+1/2+1
(9) -X+1,Y+1/2,-Z+1/2+1 (10) X,Y+1,Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H1^{1}	2.743	F1	H2^{1}	2.513
F1	H3^{2}	2.892	F1	H4^{1}	3.439
F1	H4^{2}	3.391	F1	H5B^{1}	2.714
F2	H1^{3}	2.880	F2	H2^{1}	3.093
F2	H2^{3}	2.671	F2	H3^{4}	2.459
F3	H1^{5}	2.661	F3	H2^{1}	2.591
F3	H2^{4}	3.343	F3	H3^{1}	2.795
F3	H3^{4}	3.071	F3	H4^{1}	2.400
F4	H1^{5}	2.770	F4	H4^{1}	2.989
F4	H4^{6}	2.877	F4	H5A^{6}	3.134
F4	H5A^{7}	3.039	F4	H5A^{5}	3.562
F4	H5B^{6}	2.560	C1	H3^{2}	3.440
C2	H3^{4}	3.505	C3	H1^{5}	3.176
C3	H2^{4}	3.596	C3	H3^{4}	3.559
C3	H4^{1}	3.546	C4	H1^{5}	3.488
C4	H5A^{7}	3.514	C5	H5A^{8}	3.509
C5	H5B^{8}	3.240	H1	F1^{9}	2.743
H1	F2^{3}	2.880	H1	F3^{2}	2.661
H1	F4^{2}	2.770	H1	C3^{2}	3.176
H1	C4^{2}	3.488	H1	H3^{2}	3.05
H1	H3^{10}	3.541	H1	H4^{10}	3.199
H2	F1^{9}	2.513	H2	F2^{9}	3.093
H2	F2^{3}	2.671	H2	F3^{9}	2.591
H2	F3^{11}	3.343	H2	C3^{11}	3.596
H2	H3^{11}	3.045	H3	F1^{5}	2.892
H3	F2^{11}	2.459	H3	F3^{9}	2.795
H3	F3^{11}	3.071	H3	C1^{5}	3.440
H3	C2^{11}	3.505	H3	C3^{11}	3.559
H3	H1^{5}	3.050	H3	H1^{12}	3.541
H3	H2^{4}	3.045	H3	H3^{4}	3.565
H3	H3^{11}	3.565	H4	F1^{9}	3.439
H4	F1^{5}	3.391	H4	F3^{9}	2.400
H4	F4^{9}	2.989	H4	F4^{7}	2.877
H4	C3^{9}	3.546	H4	H1^{12}	3.199
H4	H5A^{9}	3.483	H4	H5A^{7}	3.021
H5A	F4^{6}	3.039	H5A	F4^{7}	3.134
H5A	F4^{2}	3.562	H5A	C4^{6}	3.514

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H5A	C5[8]	3.509	H5A	H4[1]	3.48
H5A	H4[6]	3.021	H5A	H5A[8]	3.476
H5A	H5B[1]	3.047	H5A	H5B[8]	2.772
H5B	F1[9]	2.714	H5B	F4[7]	2.560
H5B	C5[8]	3.240	H5B	H5A[9]	3.047
H5B	H5A[8]	2.772	H5B	H5B[8]	2.906

Symmetry Operators:

(1) X,Y-1,Z
(3) -X+2,-Y+1,-Z+2
(5) X,-Y+1,Z
(7) -X+1,Y+1/2,-Z+1/2+1
(9) X,Y+1,Z
(11) -X+2,Y+1/2,-Z+1/2+1
(2) X,-Y+1,Z+1
(4) -X+2,Y+1/2-1,-Z+1/2+1
(6) -X+1,Y+1/2-1,-Z+1/2+1
(8) -X+1,-Y+1,-Z+2
(10) X,-Y+2,Z+1
(12) X,-Y+2,Z