# Polarized C-H Groups as Novel Hydrogen-Bond Donors in Hydryl-F-alkyl Esters: Unequivocal Examples for the "Pinchas Effect"

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Received July 27, 1994 (Revised Manuscript Received December 6, 1994<sup>®</sup>)

<sup>1</sup>H NMR and IR spectral evidence for a  $C-H \cdot \cdot \cdot O$  hydrogen bonds in 1-[2-hydryl-F-adamantyl] trifluoroacetate and 1,3-[2-hydryl-F-adamantyl] bis(trifluoroacetate) are presented. Evidence indicating a three-center hydrogen bond in 1,3-[2-hydryl-F-adamantyl] bis-(trifluoroacetate) is also presented. Both compounds, despite otherwise normal <sup>1</sup>H NMR chemical shift displacements and carbonyl stretching frequency depressions, show strong increases in C-H stretching frequencies related to hydrogen-bond interactions (the "Pinchas Effect").

#### Introduction

Strongly polarized C-H bonds may act as hydrogenbond donors.<sup>1</sup> Taylor and Kennard surveyed 113 neutron diffraction structures and concluded that hydrogen atoms that are covalently bonded to carbon can form intermolecular interactions which may be properly described as hydrogen bonds.<sup>2</sup> Of the potential acceptors (O, N, Cl, or S) oxygen was most conclusive. Although those studies involved intermolecular hydrogen bonding, a few examples of intramolecular hydrogen bonding were provided by the work of Michael P. Sammes and co-workers<sup>3</sup> which indicated that  $C-H \cdots N$  and  $C-H \cdots O$  hydrogen bonds exist in some substituted methylsulfonyl compounds.

Hydrogen bonding usually refers to the "two-center" (or linear) bond; however, "three-center" and "four-center" hydrogen bonds are also known.<sup>4</sup> The three-center hydrogen bond was recognized very early on, but has only been well-studied within the last decade. These studies were limited to X-ray crystallography of structures involving N-H or O-H hydrogen-bond donors.<sup>5</sup> No examples of C-H groups as hydrogen-bond donor in a three-center hydrogen-bond system have been documented, nor has any spectroscopic evidence been found in the literature.

In our work with 1-[2-hydryl-F-adamantyl] trifluoroacetate and 1.3-[2-hvdrvl-F-adamantvl] bis-trifluoroacetate. <sup>1</sup>H NMR and IR spectral evidence for  $C-H \cdot \cdot \cdot O$ hydrogen bonds are presented. Furthermore evidence for a three-center hydrogen bond in 1,3-[2-hydryl-F-adamantyl] bis-trifluoroacetate is also presented (Figure 1).

#### **Results and Discussion**

X-ray crystallography, IR spectroscopy, and <sup>1</sup>H NMR are three of the most commonly used tools for the study of hydrogen bonding. Despite their power, both X-ray



### Figure 1.

structural analysis and IR spectroscopy have some limitations. In some cases X-ray crystallography cannot distinguish a hydrogen-bond interaction from the effect of crystal-packing forces.<sup>6</sup> The infrared absorptions of the hydrogen-bonded species due to stretching vibrations of both the hydrogen-bond donor and acceptor will commonly occur at lower wavenumber than that of the unassociated species. The  $C(sp^3)$ -H bond absorption will usually shift  $10-100 (cm^{-1})$  and will increase in intensity. The C=O bond absorption will usually shift 10-50 $(cm^{-1})$ . However, the reliability of using the C-H stretching frequency as an indicator of hydrogen bonding is questionable in some cases. Lorand, et. al. have reported that in the case of  $F_2$ CHCN and  $C_6H_5$ CH(NO<sub>2</sub>)<sub>2</sub>, the C-H hydrogen-bond donor showed no change in stretching frequency.<sup>7</sup> In surprising contrast, our results show the C-H stretching vibration shifting to higher frequency!

The first observation of an increase in stretching frequency ascribed to a hydrogen bond interaction was first reported by S. Pinchas<sup>8</sup> as an explanation for the increase in aldehydic C-H stretching frequency in onitrobenzaldehydes. That interpretation was challenged by Forbes,<sup>9</sup> and by West and Whatley.<sup>10</sup> West and Whatley noted the lack of shift in the acceptor N-O stretching frequency and attributed the increase in C-H stretching frequency to steric effects analogous to those observed in 2,4,6-trimethylbenzaldehyde. In a series of articles<sup>11</sup> Pinchas argued at length that his interpretation was correct and that the increase in C-H infrared stretching frequency occurred as a result of the acute angle between donor and acceptor. The tenuousness of Pinchas's arguments have left the controversy unresolved

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#### Figure 2.

as to the nature of the interaction increasing the C-Hstretching frequency. Schneider and Bernstein<sup>12</sup> in fact observed on comparison of the infrared spectra of vapor and solid methanal an increase in the C-H stretching frequency and a decrease in the carbonyl stretching frequency on condensation of methanal (observations which are similar to those observed by us). They ascribed these shifts to a strong intermolecular association, but specifically rejected hydrogen bonding because of the directions of the C-H frequency shifts.

In support of hydrogen bonding in the 2-hydryl-Fadamantyl trifluoroacetates, we find that <sup>1</sup>H NMR offers more reliable evidence for hydrogen bonding than does IR. The fundamental difference in reliability between <sup>1</sup>H NMR and IR, according to Lorand *et. al.*, is that while the IR shift can be well defined by specific bond distance and bond angle relationships, the <sup>1</sup>H NMR chemical shift measurement takes into account all possible interactions between the hydrogen-bond donor and acceptor.<sup>7</sup>

Since hydryl-substituted perfluorinated compounds have a strongly polarized C-H bond, any hydrogen-bond acceptor which is located in a proper position may form a hydrogen bond. Our subject compounds, 1-[2-hydryl-F-adamantyl] trifluoroacetate (1) and 1,3-[2-hydryl-Fadamantyl] bis(trifluoroacetate) (2) (Figure 2) are compared to 2-hydryl-F-adamantane (3), 2-hydryl-F-adamantan-1-ol (4), 2-hydryl-F-adamantan-1,3-diol (5), and 1-F-adamantyl trifluoroacetate (6) as reference structures. Only compound 3 has been reported previously.<sup>13</sup> Compounds 1, 2, and 6 were synthesized by aerosol direct fluorination of the corresponding hydrocarbon compounds. Compounds 4 and 5 are the hydrolysis products of compounds 1 and 2, respectively.

Figure 3 shows the 250 MHz <sup>1</sup>H NMR spectrum of compounds 1, 2, 3, 4, and 5. Fluorine has the highest electronegativity of any monatomic substituent. Its strong inductive effect is responsible for the low field chemical shift of the proton on 2-hydryl-F-adamantane (3),  $\delta$  5.45 ppm, which is split into a doublet ( ${}^{2}J_{\rm H-F} =$  48.8 Hz) by the geminal fluorine atom.<sup>14</sup>

The oxygen-linked substituents have somewhat lower electronegativity than fluorine. As expected one hydroxyl group causes the hydrogen chemical shift of 1-hydroxy-2-hydryl-F-adamantane (2-hydryl-F-adamantan-1-ol) (4) to move to a relatively higher field ( $\delta = 5.23$  ppm;  ${}^{2}J_{H-F}$ = 48.2 Hz), and a second hydroxyl group causes the hydrogen chemical shift of 1,3-dihydroxy-2-hydryl-Fadamantane (2-hydryl-F-adamantan-1,3-diol) (5) to move to even higher field ( $\delta = 5.02$  ppm;  ${}^{2}J_{H-F} = 47.9$  Hz). The trifluoroacetoxyl group has an inductive effect similar to the hydroxyl group. The chemical shift of a proton adjacent to a trifluoroacetoxyl group should then be expected to occur in the same region as a proton adjacent to a hydroxyl group. Surprisingly, the proton chemical shift of 1-[2-hydryl-F-adamantyl] trifluoroacetate moves to significantly lower field ( $\delta$  6.55 ppm,  ${}^{2}J_{H-F} = 46.2$  Hz). The  $\delta$  shift to low field is 1.10 ppm. An intramolecular hydrogen bond (C-H) would be expected to have a low field shift  $(\Delta \delta)$  of about 1 ppm (in most cases less than 1 ppm).<sup>7</sup> Obviously there is a relatively strong interaction between the trifluoroacetoxyl group and the C-H group. Structure 1a (Figure 4) depicts (1) as having an intramolecular hydrogen bond which forms a six-membered ring.

The <sup>1</sup>H NMR spectrum of 1,3-[2-hydryl-*F*-adamantyl) bis(trifluoroacetate) (2) shows that the 2-hydryl chemical shift moves to even lower field ( $\delta$  = 7.70 ppm) 1.15 ppm lower than compound 1. The two bond H–F coupling constant becomes smaller (<sup>2</sup>J<sub>H-F</sub> = 43.5 Hz) than found in (1) (<sup>2</sup>J<sub>H-F</sub> = 46.2 Hz). Structure **2b** (Figure 4) shows that in **2**, two fused six-membered rings can be formed by intramolecular hydrogen bonding in this molecule. The likelihood that a three-center hydrogen bond exists is great. A linear correlation between the chemical shifts of **3**, **1**, and **2** and their two-bond coupling constants, <sup>2</sup>J<sub>H-F</sub> is shown in Figure 5.

The linear correlation in Figure 5 has a precedent. Evans discovered that HCBrClF forms hydrogen bonds with different solvents.<sup>15</sup> In his measurements the twobond H-F coupling constant in HCBrClF exhibited a linear relationship with the chemical shift variation due to each different solvent. The reason the  ${}^{2}J_{H-F}$  changed with the different hydrogen-bond acceptors is not quite clear. In our systems, the excellent correlation found between the  ${}^{2}J_{H-F}$  and the chemical shift of the reference species 3 and the two-center 1 and three-center 2 hydrogen-bonded species (Figure 2) seems clearly related to the number of hydrogen-bond acceptors. We do not have a rigorous physical explanation, but the correlation is quite clear. A correlation which is not observed between compounds 3, 4, and 5 (Table 1).

The IR spectral data are listed in Table 2. The C=O stretching frequency of 1-*F*-adamantyl trifluoroacetate (6) is 1848 cm<sup>-1</sup>. The C=O stretching frequency of 1-[2-hydryl-*F*-adamantyl] trifluoroacetate (1) is found to shift to a lower frequency, 1829 cm<sup>-1</sup>;  $\Delta \nu$  is 19 cm<sup>-1</sup>. This shift to lower wavenumber is also found in 1,3-[2-hydryl-*F*-adamantyl] bis(trifluoroacetate) (2). The frequency is shifted to 1832 cm<sup>-1</sup>;  $\Delta \nu$  is 16 cm<sup>-1</sup> relative to (6).

The decrease in the C=O stretching frequency of compound 2 was less than that of compound 1. In the two-center hydrogen bond, the interaction is only between

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<sup>(14)</sup> Further splitting into a quintet and additional lower order splitting in Figure 5 are due to "long-range virtual coupling". This is a common splitting phenomenon appearing in polyfluorinated compounds especially in polyfluorinated adamantanes. A detailed discussion is beyond the scope of this paper. For further information about virtual coupling, see Musher, J. I.; Corey, E. J. Tetrahedron 1962, 18, 791.

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Figure 3.







Figure 5. Correlation of chemical shift ( $\delta$  in ppm) and coupling constant ( ${}^{2}J_{\rm H-F}$  in hertz).

one hydrogen-bond donor and one acceptor. In the threecenter hydrogen bond, the interaction with the hydrogenbond donor is split between the two acceptor carbonyls.

Table 1. Proton Chemical Shift and  ${}^{2}J_{H-F}$  of compounds

1-5		
δ (ppm)	$^{2}J_{\mathrm{H-F}}\left(\mathrm{Hz} ight)$	
5.02	47.9	
5.23	48.2	
5.45	48.8	
6.55	46.2	
7.70	43.5	
	1-5           δ (ppm)           5.02           5.23           5.45           6.55           7.70	

 

 Table 2.
 C=O and C-H Stretching Frequency of Compounds 1-3 and 6<sup>a</sup>

IR	$\nu_{\rm C=0}~({\rm cm}^{-1})$	$\nu_{\rm C-H}  ({\rm cm}^{-1})$
3		2986
6	1848	
1	1829	3020
2	1832	3051

<sup>a</sup> Thin film between KCl disks.

For the same hydrogen-bond donor and acceptor, the three-center hydrogen-bond energy would be expected to be weaker than twice the two-center hydrogen-bond energy. It seems reasonable that the one C=O stretching frequency in compound (1) will move to lower wavenumber than the two C=O's of compound 2.

The C-H stretching frequency results are surprising. Instead of moving to lower wavenumber absorptions in both compounds 1 and 2, they move to higher wavenumber and increase in intensity relative to compound 3 (Figure 6). Compound 3 has a C-H stretching frequency of 2986 cm<sup>-1</sup> while that of 1 is 3020 cm<sup>-1</sup>, and that of 2 is 3051 cm<sup>-1</sup>. From the values of  $\Delta v_s / \Delta v$  it seems clear that the FC-H···O = C are relatively weak compared to O-H···O = C hydrogen bonds. However the  $\Delta v_{C-O}$  are 19 cm<sup>-1</sup> and 16 cm<sup>-1</sup>, respectively, for 1 and 2 and relative to their  $\Delta v_s$  C-H values of 34 and 65 cm<sup>-1</sup> are relatively much larger and in the opposite direction to the  $\Delta v_{N=O}$  values cited by Pinchas<sup>11c</sup> and by West and Whatley<sup>10</sup> in the case of o-nitrobenzaldehyde.



## Figure 6.

It seems clear that the FC-H···O = C hydrogen bond is bent in 1 and probably more so in 2. Whether this has anything to do with the increase in the  $\nu_s$  C-H between these two compounds is not clear; however, molecular models suggest that the C-H points to one of the lone-pairs on the oxygen (proton) acceptor in 1 and toward a region of two lone-pairs, one from each of the carbonyl oxygens in 2. Since the  $\Delta \nu_s$  C-H increases with this presumed interaction, an electric field effect may be operative, especially since the bent hydrogen bond will, with greater deviations from 180°, minimize the effect of multicenter bonding in lowering the C-H bond order.

## **Experimental Section**

<sup>19</sup>F NMR spectra were recorded on a JEOL FX90Q FTNMR operating at 84.7 MHz, using CFCl<sub>3</sub> as solvent and internal standard. <sup>1</sup>H NMR spectra were recorded on a Bruker AC250 MHz instrument using CFCl<sub>3</sub>/CDCl<sub>3</sub> mixture (1:3, v/v) as solvent. CFCl<sub>3</sub> is necessary to dissolve the fluorinated compounds and CDCl<sub>3</sub> is used for the internal field-frequency lock. IR spectra were recorded on a Bio-Rad Spc3200 FTIR spectrophotometer. Negative chemical ionization (electron attachment) mass spectra were recorded on a VG. ZAB-EQ mass spectrometer. Samples were introduced into the source via the reference inlet to a pressure of  $10^{-6}$  torr and diluted with nitrogen gas to  $10^{-5}$  to  $10^{-4}$  torr and bombarded with 70 eV electrons. High resolution mass spectra were recorded on the VG ZAB-EQ mass spectrometer in positive ion EI mode at 70 eV. All gas chromatography is run on a Fluorosilicone QF-1 column.

1-Adamantanol is commercially available from Aldrich. 1,3-Adamantanediol was synthesized by literature methods.<sup>16</sup>

1-Adamantyl Acetate (7). A mixture of 3 g of 1-adamantanol and 20 mL of acetyl chloride was stirred at 0 °C for 1 h and at room temperature for 2 h. After evaporation of excess acetyl chloride, 4.0 g crude product was obtained, containing 59% of (7) and 41% of unreacted starting material. Separation was effected by column chromatography using hexane/acetyl acetate (20:1) as eluent. Characterization of 7: <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$  1.62 (m, 6H), 1.88 (s, 3H), 2.01–2.06 (m, 9H); <sup>13</sup>C NMR (CDCl<sub>3</sub>)  $\delta$  170.2, 80.2, 41.3, 36.2, 30.8, 22.6; mp 31–32 °C, literature mp 33 °C.<sup>17</sup>

1,3-Adamantyl Diacetate (8). A mixture of 2.5 g of 1,3adamantanediol and 50 mL of acetyl chloride was stirred at 0 °C for 15 min, at room temperature for 1 h, under reflux condition for 2 h and while cooling to room temperature for another 1 h. After evaporation of excess acetyl chloride, 3.2 g of crude product was obtained, containing 82% of 8 and 18% unreacted starting material. Separation was effected by column chromatography using hexane/acetyl acetate (10:3) as eluent. Characterization of 8: <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$  2.30 (s, 2H), 2.18 (s, 2H), 1.92 (s, 8H), 1.82 (s 6H), 1.42 (s, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>)  $\delta$  169.7, 80.0, 44.9, 39.7, 34.5, 30.8, 22.1. The <sup>1</sup>H NMR is consistent with published data.<sup>18</sup>

**Aerosol Fluorination.** Compounds 7 and 8 were injected into the reactors as solutions in a cofluorinating solvent; detailed procedures are described elsewhere.<sup>19</sup> Specific reaction parameters are listed in Table 3 (supplementary material).

Aerosol Fluorination of 7. 7 (2.4 g) was dissolved in 7.5 g of CH<sub>2</sub>ClCHCl<sub>2</sub>. After the injection (Table 3) was finished, the crude product was transferred onto a vacuum line and fractionated at trap temperature of -22 °C, -45 °C, -78 °C, -131 °C, and -196 °C. The major products were collected in the -22 °C trap with some perfluorinated solvent. GC analysis shows two major products (50:50) (column temperature program: 60 °C for 12 min; 30 °C/min to 150 °C, 25 min). Preparative GC affords pure 6 (0.47 g, 9.4% yield) and pure 1 (0.54 g, 10.2% yield).

Characterization of **6**: <sup>19</sup>F NMR (CFCl<sub>3</sub>)  $\delta$  -74.4 (s, 3F), -114.6 (s, 6F), -121.1 (s, 6F), -221.7 (s, 3F); IR (film)  $\nu_{C=O}$ 1848 cm<sup>-1</sup>. Further characterization is provided by characterization of the hydrolysis product of **6**, 1-*F*-adamantanol:<sup>20</sup> MS (EI); *m/z* (inten, ident) 422 (17, M<sup>+</sup>); 403 (24, M<sup>+</sup> - F); 203 (100); <sup>19</sup>F NMR (CFCl<sub>3</sub>)  $\delta$  -123.0 (s, 12F), -224.3 (s, 3F).

Characterization of 1:  $^{19}$ F NMR (CFCl<sub>3</sub>)  $\delta$  –74.9 (s, 3F), –111.7 to –126.2 (m, 10F), –212.5 (s, 1F), –221.6 to –223.0 (m, 3F);  $^{1}$ H NMR (CDCl<sub>3</sub>)  $\delta$  6.55 (dq,  $^2J$  = 46.2 Hz); IR  $\nu_{\rm C-H}$  3021 cm<sup>-1</sup>,  $\nu_{\rm C=0}$  1829 cm<sup>-1</sup>. Further characterization is provided by characterization of the hydrolysis product of 1, 2-hydryl-F-1-adamantanol (4):<sup>20</sup> HRMS C<sub>10</sub>H<sub>2</sub>F<sub>14</sub>0 calcd 403.98821, expt 403.9874; MS (EI) m/z (inten, ident) 404 (63, M<sup>+</sup>); 385 (30, M<sup>+</sup> – F); 203 (100); EA 404 (1, M<sup>1–</sup>), 403 (3, M<sup>1–</sup> – 1), 384 (100, M – HF);  $^{19}$ F NMR (CFCl<sub>3</sub>)  $\delta$  –116.4 to –127.1 (m, 10F), –213.7 (s, 1F), –222.5 to –226.3 (m, 3F); <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$  5.23 (dq,  $^2J$  = 48.2 Hz); IR (film) 3379 (broad), 2994 cm<sup>-1</sup> (w).

Aerosol Fluorination of 8. 8 (1.5 g) was dissolved in 4.6 g of CH<sub>2</sub>ClCHClCH<sub>2</sub>Cl. The solution injection (Table 3) and other procedures were similar to the fluorination of 7. GC analysis showed one major product (column temperature program: 80 °C for 12 min; 30 °C/min to 180 °C, for 25 min). Preparative GC afforded pure 2 (0.16 g, 5.0% yield). Characterization of 2: <sup>19</sup>F NMR (CFCl<sub>3</sub>)  $\delta$  -74.9 (s, 6F), -112.0 to -121.3 (m, 10F), -218.8 to -221.4 (m, 3F); <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$  7.70 (dt, <sup>2</sup>J = 43.5 Hz); IR (film)  $\nu_{\rm C-H}$  3051,  $\nu_{\rm C-O}$  1832 cm<sup>-1</sup>. Further characterization is provided by characterization of the hydrolysis product of  ${\bf 2},$  compound  ${\bf 5}^{:20}\,HRMS\,C_{10}H_3F_{13}O_2$  calcd 401.9926, expt 401.9919; MS (EI) m/z (inten, ident) 402 (40, M<sup>+</sup>); 383 (12, M<sup>+</sup> - F); 69 (100, CF<sub>3</sub>). <sup>19</sup>F NMR (CFCl<sub>3</sub>)  $\delta$ -120.3 to -123.9 (m, 10F), -221.9 to -225.7 (m, 3F); <sup>1</sup>H NMR  $(\text{CDCl}_3) \delta 5.02 \text{ (dt, } {}^2J = 47.9 \text{ Hz}); \text{ IR (film) } 3385 \text{ (broad), } 2970$  $cm^{-1}$  (w).

**Supplementary Material Available:** Specific aerosol direct-fluorination parameters (Table 3), complete IR spectra of 1, 2, 3, and 6 (5 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

#### JO941297K

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<sup>(20)</sup> A detailed hydrolysis study will appear in a future publication.