stirrer. Then **4**-MeCN or **5**-H_2O was added, and the resultant solution was stirred intermittently. Runs with **2** as substrate were performed analogously and were monitored by the appearance of **1a** at 288 nm (λ_{max}); A_∞ was determined by experiment. A MeCN solution was used for its addition to HTABr–buffer. In no run was there evidence for hydrolytic cleavage of the phosphonium salt as found[11] in other systems at higher pH.

pK_a of 1. The pK_a of **1** was measured by UV[3] and ^{31}P NMR methods. In the former, three buffers containing 0.002 M HTABr were prepared at pH 2.0 (0.013 M HCl, μ = 0.13 (KCl)), 7.0 (0.06 M phosphate, μ = 0.13), and 12.0 (0.025 M phosphate, μ = 0.13). To each **1**-MeCN was added to give 1.0×10^{-4} M **1** and 0.4% (v/v) MeCN. Absorbances (A) were measured for each solution at 25 °C at 5-nm intervals over the range of 240–300 nm. The ratio $(A_{7.0} - A_{2.0})/(A_{12.0} - A_{7.0})$ was taken as $[A^-]/[HA]$ and was calculated at each wavelength. An average value of 3.81 was obtained (with exclusion of anomalous data from 260–280 nm) to give pK_a = 7.0 – log 3.81 = 6.4. In the NMR method, three buffers containing 0.01 M HTABr were prepared at pH 2.0 and 7.0 (same as above) and at 10.8 (0.0125 M borate, μ = 0.13 (KCl)). To each **1**-MeCN was added to give 0.01 M **1** and 2.2% (v/v) MeCN. ^{31}P NMR chemical shifts of 19.45, 18.50, and 17.92, respectively, were obtained. The ratio $[A^-]/[HA]$ was determined from $\delta_{7.0} = X_{A^-}\delta_{10.8} + X_{HA}\delta_{2.0}$ and $X_{A^-} + X_{HA} = 1$, wherein X = mole fraction. A value of 1.63 was obtained to give pK_a = 7.0 – log 1.63 = 6.8. The average pK_a (6.6) from the UV and NMR methods was used in the text.

Acknowledgment is gratefully made to the U.S. Army Research Office and the Marathon Oil Company.

Registry No. **1**, 100466-58-8; **2**, 100466-59-9; **3**, 100466-60-2; **4**, 830-03-5; **5**, 4134-83-2; HTABr, 57-09-0; p-$MeOC_6H_4PPh_2$, 896-89-9; n-$C_{16}H_{33}Br$, 112-82-3.

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Unexpected Formation of 1,4,7,9-Tetrafluorodiamantane in the Reaction of 1,4,9-Tribromodiamantane with $NO_2^+BF_4^-$/Pyridinium Polyhydrogen Fluoride (PPHF)

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Recently we reported[1] the preparation and NMR spectroscopic (^{13}C and ^{19}F) study of a series of bridgehead mono- and polyfluorinated adamantanes and diamantanes. The bridgehead fluorides were conveniently prepared in excellent yields from the corresponding bromo compounds using $NO_2^+BF_4^-$ pyridinium polyhydrogen fluoride (PPHF).[2] In that study we reported the preparation of two bridgehead difluorodiamantanes (namely, 1,6-difluorodiamantane and 4,9-difluorodiamantanes). The similar conversion of adamantanoid halides to fluorides was also possible by the action of elemental fluorine.[3] In continuation of our studies we were interested in preparing the tri- and tetrafluorodiamantanes from the corresponding bromo compounds. We now report our results in the reaction of 1,4,9-tribromodiamantane,[4] (**1**) with excess $NO_2^+BF_4^-$/PPHF reagent which gave, unexpectedly, 1,4,7,9-tetrafluorodiamantane (**2**).

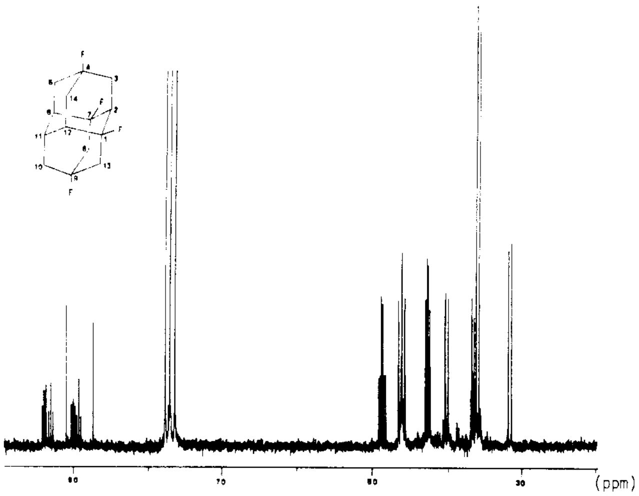
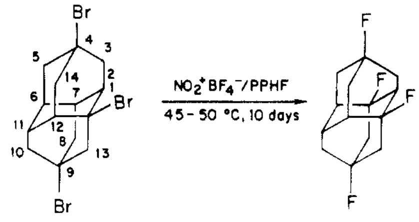


Figure 1. ^{13}C NMR spectrum of 1,4,7,9-tetrafluorodiamantane (**2**) in $CDCl_3$ at room temperature.



1 **2**

Results and Discussion

1,4,9-Tribromodiamantane (**1**) was prepared according to literature procedure[4] by ionic bromination of diamantane. The elemental analysis and ^{13}C NMR spectral characteristics [$\delta^{13}C$: 70.2 (s), 60.2 (s), 59.6 (s), 59.3 (t), 48.6 (t), 47.8 (d), 47.3 (t), 44.7 (t), 41.9 (d), 38.2 (d)] correspond well with the structure, and the melting point (190–192 °C) is very close to that reported in the literature[4b] (193–195 °C).

Reaction of **1** with excess $NO_2^+BF_4^-$ in 70% pyridinium polyhydrogen fluoride (PPHF) at 45–50 °C in an autoclave for 10 days gave in 72% yield a tetrafluorocompound with molecular formula $C_{14}H_{16}F_4$ as determined by elemental analysis (see Experimental Section for details). ^{19}F NMR[5] of the product showed three signals at $\delta(^{19}F)$ –142.9 (1 F), –146.8 (1 F), and –150.4 (2 F). These are in the region of bridgehead ^{19}F chemical shift.[1] Also, the absence of any observable ^{19}F–^{1}H coupling indicates that all the fluorine atoms are at the bridgehead position. The ^1H noise-decoupled ^{13}C NMR spectrum in $CDCl_3$ is shown in Figure 1. It shows ten ^{13}C multiplets (^{13}C–^{19}F coupling), indicating a C_s symmetry in the diamantane skeleton. The ^{13}C chemical shifts along with their proton multiplicites (obtained using APT experiment[6]) are listed in the Table I.

The spectral characteristics (both ^{13}C and ^{19}F) indicate that the product is 1,4,7,9-tetrafluorodiamantane (**2**). Every individual ^{13}C–^{19}F multiplet in the ^{13}C NMR spec-

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(1) Olah, G. A.; Shih, J. G.; Krishnamurthy, V. V.; Singh, B. P. *J. Am. Chem. Soc.* **1984**, *106*, 4492.

(2) (a) Olah, G. A.; Shih, J. G.; Singh, B. P.; Gupta, B. G. B. *Synthesis* **1983**, 713. (b) The present method of ionic fluorination is well suited for tertiary and adamantanoid halides but seems to be limited to those tertiary alkyl halides which cannot undergo olefin-forming elimination.

(3) Rozen, S.; Brand, M. *J. Org. Chem.* **1981**, *46*, 733.

(4) (a) Gund, T. M.; Schleyer, P. v. R. *Tetrahedron Let.*. **1971**, 1583. (b) Gund, T. M.; Schleyer, P. v. R.; Unruh, G. D.; Gleicher, G. J. *J. Org. Chem.* **1974**, *39*, 2995.

(5) ^{19}F chemical shifts are with respect to $CFCl_3$. Negative sign indicates shielding.

(6) LeCoeq, C.; Lallemand, J. Y. *J. Chem. Soc., Chem. Commun.* **1981**, 150.

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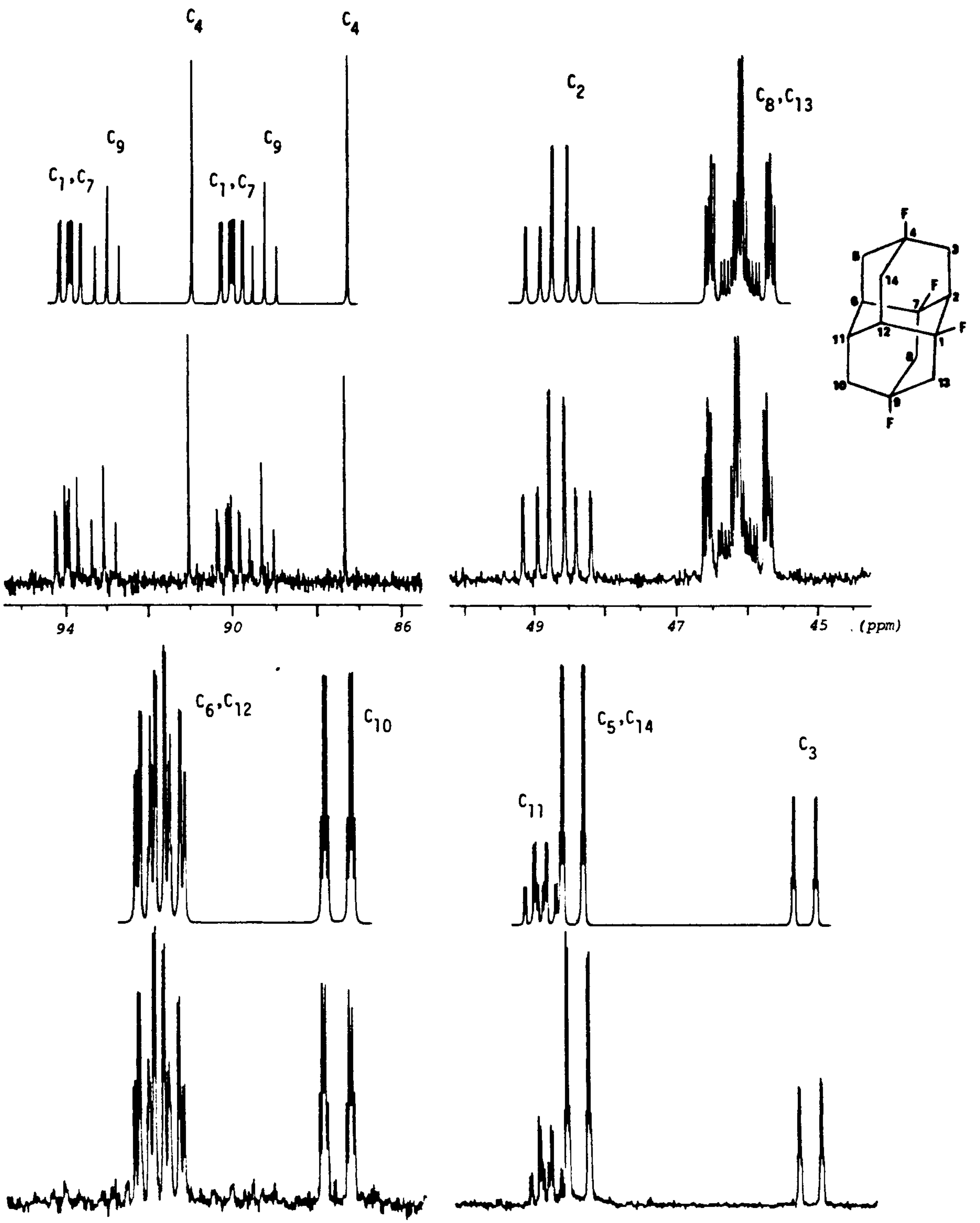

Figure 2. Experimental (lower traces) and simulated (upper traces) ^{13}C NMR multiplets in 1,4,7,9-tetrafluorodiamantane (2).

trum of 2 was analyzed by spin simulation followed by spectral fitting with the experimental spectrum using the program LAME in the iterative mode.[7,8] Initial estimates of the coupling constants (J_{CF} and J_{FF}) were based on the values reported earlier[1] for fluoroadamantanes and mono- and difluorodiamantanes. The chemical shifts and the coupling constants were iterated[9] starting with these initial values by using the experimental spectrum as reference. Iterations were continued until the root-mean-square (rms) frequency error reached a minimum. The calculated spectrum (for each individual multiplet) is shown in Figure 2 along with the experimental spectrum. It must be noted

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[7] The spin simulation is based on the Fortran program LAME, which is LAOCOON with magnetic equivalence added.[8] LAME calculates the theoretical spectrum for spin = $^1/_2$ nuclei given the values of chemical shifts and coupling constants. It can adjust the values of the parameters to approach a given experimental spectrum.

[8] (a) Bothner-by, A. A.; Castellano, S. *J. Chem. Phys.* **1964**, *41*, 3863. (b) Emsley, J. W., Feeney, V., Sutcliffe, L. M., Eds.; "Progress in Nuclear Magnetic Resonance Spectroscopy"; Pergamon Press: Oxford, 1966% Vol. 1, Chapter 3. Stanley, R. M.; Marquardt, D. W.; Ferguson, R. C. *J. Chem. Phys.* **1964**, *41*, 2087.

[9] Iterations were done alternatively on the coupling constants and the chemical shifts by keeping the other constant. This was continued until a minimum in the RMS frequency errors (usually less than 0.2 Hz) was achieved.

Table I. ^{13}C and ^{19}F NMR Parameters for 1,4,7,9-Tetrafluorodiamantane in $CDCl_3$

carbon no.	$\delta(^{13}C)^a$	$^{13}C-^{19}F$ NMR coupling constants^b (Hz)	$\delta(^{19}F)^c$
C_1, C_7	92.0, s	$^1J_{C_1F_1} = 194,9$; $^3J_{C_1F_7} = 11.1$; $^3J_{C_1F_9} = 14.7$; $^4J_{C_1F_4} = 2.0$	$\delta_{F_1(F_7)} = -150.4$
C_9	91.5, s	$^1J_{C_9F_9} = 190.2$; $^3J_{C_9F_1} = 14.3$; $^7J_{C_9F_4} \approx 0$	$\delta_{F_9} = -146.3$
C_4	89.2, s	$^1J_{C_4F_4} = 185.1$; $^4J_{C_4F_1} \approx 0$; $^7J_{C_4F_9} \approx 0$	$\delta_{F_4} = -142.9$
C_2	48.6, d	$^2J_{C_2F_1} = 19.1$; $^3J_{C_2F_4} = 10.7$; $^4J_{C_2F_9} = 0.8$	$^{19}F-^{19}F$ NMR coupling constant^d
C_8, C_{13}	46.1, t	$^2J_{C_8F_7} = 20.8$; $^2J_{C_8F_9} = 19.8$; $^4J_{C_8F_1} = -1.3$; $^5J_{C_8F_4} = 2.3$	$^4J_{F_1F_9} = 8.2$; $^4J_{F_1F_7} = 6.6$
C_6, C_{12}	42.5, d	$^2J_{C_6F_7} = 19.0$; $^3J_{C_6F_4} = -10.9$; $^4J_{C_6F_1} = -1.5$; $^4J_{C_6F_9} = -1.3$	$^5J_{F_1F_4} = 0.4$; $^7J_{F_4F_9} = 2.0$
C_{10}	40.0, t	$^2J_{10}F_9 = 18.9$; $^4J_{C_{10}F_1} = 1.7$; $^5J_{C_{10}F_4} = 1.0$	$\Delta\delta_{FF}(^{13}C)^{e,12}$
C_{11}	36.5, d	$^3J_{C_{11}F_1} = 8.7$; $^3J_{C_{11}F_9} = 11.7$; $^4J_{C_{11}F_4} = 1.7$	$\Delta\delta_{FF}(^{13}C_8) = 2.9$ Hz (-0.016 ppm)
C_5, C_{14}	35.9, t	$^2J_{C_5F_4} = 20.3$; $^3J_{C_5F_7} = -1.8$; $^5J_{C_5F_1} = 0.9$; $^5J_{C_5F_9} = 1.3$	$\Delta\delta_{FF}(^{13}C_6) = -2.7$ Hz (-0.015 ppm)
C_3	31.6, t	$^2J_{C_3F_4} = 21.2$; $^3J_{C_3F_4} = 1.2$; $^5J_{C_3F_9} = 1.2$	$\Delta\delta_{FF}(^{13}C_5) \simeq -0.1$ Hz (0.0 ppm)

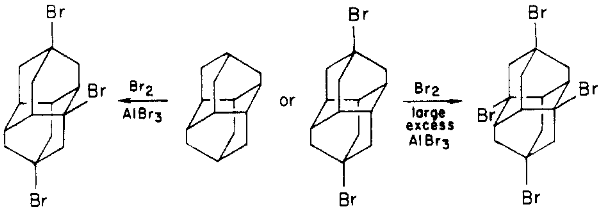
[a] ^{13}C chemical shifts are in ppm (±0.05) with respect to external Me_4Si in $CDCl_3$ at room temperature (~20 °C); s = singlet, d = doublet, t = triplet. These represent carbon-proton multiplicities obtained using APT experiment.[6] [b] All coupling constants are in hertz and are accurate to ±0.5 Hz. They were either measured directly in the case of simple multiplets or calculated using spin simulation technique. Signs of the coupling constants, whereever indicated, are based on their relative sign with respect to ^2J which was assumed to be positive.[17] [c] ^{19}F chemical shifts are in ppm (±0.1) with respect to external $CFCl_3$ in $CDCl_3$ at room temperature (~20 °C); negative sign indicates upfield shift. [d] Coupling constants are in hertz and are accurate to ±0.3 Hz. They were calculated by spin simulation technique. [e] The chemical shift differences are between F_1 and F_7 and are given both in hertz (±0.3) (at 188.2 MHz ^{19}F resonance frequency) and in ppm.

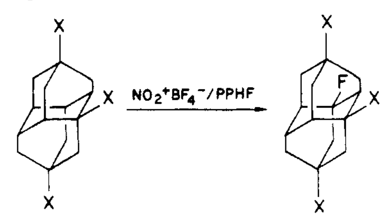
that C_6 (or C_{12}) and C_8 (or C_{13})[10] show complex multiplicity patterns due to the fact that they will induce differential ^{13}C isotope effects on F_1 and F_7 chemical shifts[1,11] whereas all other non-fluorinated carbons (i.e., C_2, C_3, C_{10}, and C_{11}) are symmetrically placed between F_1 and F_7 and thus appear as simple multiplet. One would expect a complex pattern in the ^{13}C resonance of C_1. The $^{13}C_1$ is also not equivalently located with respect to F_1 and F_7 and thereby should introduce differential ^{13}C isotope effects on F_1 and F_7 chemical shifts. However, the coupling pattern of the C_1 carbon resonance is deceptively simple (a doublet of a doublet of a doublet of a doublet), similar to the simple pattern observed for C_1 resonance in 1,3-difluoroadamantane and 1,3,5,7-tetrafluoroadamantane.[1] The C_9 resonance does not show resolvable coupling with F_4 and thus appears as a doublet of triplets, while the C_4 resonance does not show resolvable coupling with F_1, F_7, and F_9 and appears as a simple doublet.

All the $^{13}C-^{19}F$ NMR coupling constants were calculated from the spin similation technique (or measured from the experimental spectrum where the coupling pattern is simple) and are listed in the table along with ^{13}C chemical shifts. The $^{13}C-^{19}F$ coupling constants in **2** are close to what one would expect based on our earlier report.[1] The $^{19}F-^{19}F$ NMR coupling constants (calculated values) are $^4J_{F_1F_7} = 6.6 \pm 0.3$ Hz, $^4J_{F_1F_9} = 8.2 \pm 0.3$ Hz, and $^5J_{F_1F_4} = 0.4$ Hz. The $^4F_{FF}$ values are close to those reported in di-, tri-, and tetrafluoroadamantanes. The ^{13}C isotope-induced F_1 and F_7 chemical shift differences $[\eta\delta_{FF}(^{13}C)]$[12,13] are $\Delta\delta_{FF}(^{13}C_8) = -0.016$ ppm, $\Delta\delta_{FF}(^{13}C_6) = -0.015$ ppm, and $\Delta\delta_{FF}(^{13}C_5) \simeq 0$ ppm. C_8 and C_6 being β to one fluorine and δ to the other introduce similar isotope effects. These isotope effects are, again, close to those observed in di-, tri-, and tetrafluoroadamantanes.[1] C_5 being γ_{syn} to F_7 and ϵ to F_1 introduces negligible ^{13}C isotope effect.

Thus, the product of the reaction of 1,4,9-tribromodiamantane with $NO_2^+BF_4^-$/PPHF is clearly 1,4,7,9-tetrafluorodiamantane. The fluorine atoms at 1,4, and 9 positions are obviously introduced through halogen exchange reaction with nitronium ion acting as bromide abstracting agent. The ionic C-H fluorination of the C_7 position in the $NO_2^+BF_4^-$/PPHF system is not unprecedented. For example, adamantane reacts readily with $NO_2^+BF_4^-$ in PPHF to give 1-fluoroadamantane.[14]

In the present study although the sequence of fluorination has not been established, the fluorination at the C_7 position clearly occurred on a 1,4,9-trihalodiamantane. This result is rather suprising based on the fact that ionic bromination of diamantane or 4,9-dibromodiamantane with Br_2 catalyzed by a large excess of $AlBr_3$ gives 1,4,6,9-tetrabromodiamantane.[4] With limited quantities of $AlBr_3$, 1,4,9-tribromodiamantane is isolated in the reaction of 4,9-dibromodiamantane and thus must be the intermediate in the formation of the tetrabromide with excess $AlBr_3$. In other words, ionic bromination of 1,4,9-tribromodiamantane with Br_2 and excess $AlBr_3$ occurs at the C_6 position which is δ to two bromine (C_1-Br and C_9-Br) atoms and γ to the third bromine (C_4-Br). However, in the present study ionic fluorination of 1,4,9-trihalodiamantane occurs at C_7 which is γ to two halogens (C_1-X and C_4-X) and δ to the other halogen (C_9-X). One can argue that a carbocationic center developed at the C_7 position (by hydride abstraction) in 1,4,9-trihalodiamantanes derives more stability from the lone pairs on C_1-X and C_9-X halogens through C_1-C_2 and C_8-C_9 σ-bond hyperconjugation, as depicted in **3**, whereas a carbocationic center developed at the C_6 position derives stability only from the lone pairs on C_4-X through C_4-C_5 σ-bond hy-





X = F or Br

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[10] One would expect C_5 (or C_{14}) also to show complex pattern. But the calculation by spin simulation indicates that ^{13}C isotope-induced ^{19}F chemical shift difference is less than 0.1 Hz and thus appear as a simple multiplet. This small $\Delta\delta_{FF}(^{13}C)$[12] is to be expected as C_5 is γ_{syn} to F_7 and ϵ to F_1. Even ^{13}C isotopes that are γ_{syn} to one fluorine and δ to another fluorine induce only 0.5-Hz isotope effect.

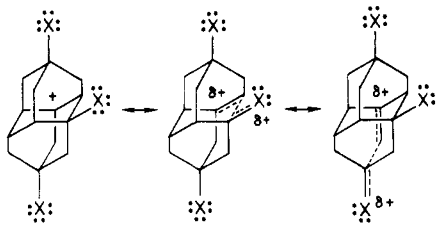
[11] Weigent, F. J.; Robest, J. D. *J. Am. Chem. Soc.* **1971**, *93*, 2361.

[12] $\Delta\delta_{FF}(^{13}C)$ is defined as δ_F(nearer to ^{13}C) − δ_F(farther from ^{13}C) and are in general negative, as ^{13}C isotope shields a fluorine nucleus compared to ^{12}C isotope.[13]

[13] Batiz-Hernandez, H.; Bernheim, R. A. "Progress in Nuclear Magnetic Resonance Spectroscopy", Emsley, J. W.; Feeney, V.; Sutcliff, L. M. Eds.; Pargamon Press: New York, 1967; Vol. 3, Chapter 2.

[14] Olah, G. A.; Shih, J. G.; Singh, B. P.; Gupta, B. G. B. *J. Org. Chem.* **1983**, *48*, 3356.

perconjugation as in 4. Such C–C hyperconjugative stabilization in adamantyl and diamantyl cations under superacid conditions is well established.[15] Thus, the product observed in PPHF is the one with fluorine at C_7 position. In the quoted study[4] on ionic bromination, it is quite likely that the initially formed 1,4,7,9-tetrabromodiamantane in the presence of excess $AlBr_3$ rearranges to the more symmetrical and probably thermodynamically more stable 1,4,6,9-tetrabromodiamantane. This would explain that under the preparative conditions employed in the study[4] no 1,4,7,9-tetrabromodiamantane was detected.



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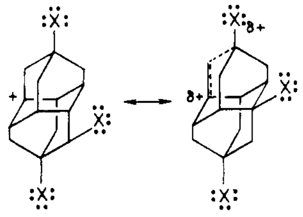

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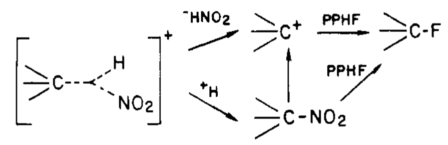
Concerning the mechanism of the fluorinations, NO_2^+ is capable of bringing about bromide abstraction from the C–Br bonds followed by quenching of the bridgehead carbocations by fluoride from the pyridinium polyhydrogen fluoride reagent. This accounts for the S_N1 type fluorine exchange reaction of 1,4,9-tribromodiamantane. The nitronium ion is, however, also capable of attacking the tertiary bridgehead C–H bond at C_7. The transition state must involve 2e–3c interaction. No nitrodiamantane products were, however, observed, indicating that even if nitration (competing with hyrogen abstraction) would take place, the tertiary bridgehead nitrocompounds would cleave to the same fluorinated product. Indeed electrophilic nitration of diamantane ith $NO_2^+BF_4^-$ can be carried out in solvent media other than PPHF and will be reported subsequently.



Experimental Section

1,4,9-Tribromodiamantane (1) was prepared from diamantane[16] with a literature procedure.[4] The compound gave satisfactory elemental and spectral analyses.

Reaction of 1,4,9-Trifluorodiamantane (1) with NO_2^+-BF_4/PPHF. To a solution of 5 g (38 mmol) of $NO_2^+BF_4^-$ in 100 mL of pyridinium polyhydrogen fluoride[2] (70%) in a 250-mL pressure reaction vessel at 0 °C was added 3.15 g (7.5 mmol) of 1,4,9-tribromodiamantane (1) with continuous stirring. After the addition the reaction mixture was stirred at 40–45 °C for 10 days. The mixture was poured into crushed ice and extracted with (2 × 100 mL) ether. The organic layer was washed successively with water, saturated $NaHCO_3$ solution, and finally with brine and dried over $MgSO_4$ and the solvent was distilled at low pressure. The 1,4,7,9-tetrafluorodiamantane (2) obtained was >90% pure and was further purified by chromatography on a silica gel column with hexane–chloroform as eluant. The yield of the purified product is 1.4 g (5.4 mmol, 72%). An analytical sample was prepared by sublimation over a steam bath at reduced pressure (~20 torr). It gave satisfactory elemental analysis (Found: C, 64.36; H, 6.15; F, 29.39. Calcd: C, 64.61; H, 6.20; F, 29.20.): mp 274–276 °C. The ^{13}C and ^{19}F NMR spectral data are given in Table I.

The ^{13}C NMR spectra were recorded in $CDCl_3$ (which also acts as the deuterium source for internal field-frequency lock) solution at room temperature on a Varian XL-200 superconducting NMR spectrometer at 50.3 MHz ^{13}C operating frequency with broad-band proton noise decoupling. Chemical shifts are referenced to external Me_4Si, and the assignments are based on their position, carbon–fluorine coupling pattern, and magnitude, and proton multiplicites are obtained using APT (attached proton test)[6] experiment. The ^{19}F spectrum was recorded in $CDCl_3$ solution at room temperature on a Varian XL-200 NMR spectrometer at 188.2 MHz ^{19}F operating frequency. The chemical shifts are referenced to external $CFCl_3$ and are accurate to ±0.1 ppm.

Spin simulation and spectral fitting are based on standard Fortran program, LAME.[7,8] Initial guesses of $^nJ_{CF}$ and J_{FF} were made with the values reported earlier.[1] The values of $^nJ_{CF}$, J_{FF}, and $\Delta\delta_{FF}(^{13}C)$ were adjusted by trial to approach the experimental spectrum within 1–2 Hz. The coupling constants and the chemical shifts were then iterated to approach the given experimental spectrum. The iterations were continued until the root-mean-square (rms) frequency error reached a minimum. In general, rms frequency errors at the end of the iterations were <0.2 Hz with respect to the given experimental spectrum. The relative signs of the coupling constants were based on the initial assumption that $^2J_{CF}$ is positive.[17] The fluorine nearer to a ^{13}C isotope was always assumed to be more shielded compared to the one farther to ^{13}C.[13] In other words, $\Delta\delta_{FF}(^{13}C)^{12}$ was always assumed to be negative.

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Registry No. 1, 32401-13-1; 2, 100466-62-4.

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The Conformational Preference of the Diphenylphosphinoyl Group in Cyclohexane[1]

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The discovery of a predominant *axial* conformation in [2-(1,3-dithianyl)]diphenylphosphine oxide (1, eq 1) was