Chemistry of Organo Halogenic Molecules. XLVIII. Stereochemistry of Halofluorination of 1,2- and 1,4-Dihydronaphthalene

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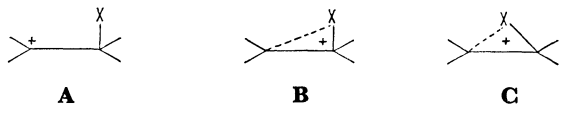
Halofluorination of 1,2-dihydronaphthalene with a mixture of *N*-chlorosuccinimide or *N*-bromosuccinimide or *N*-iodosuccinimide–hydrogen fluoride–pyridine in ether proceeds with Markovnikov type regioselectivity. The reaction is stereospecifically anti, the anti adduct isomerizing to syn products under the reaction conditions. The elimination of hydrogen halide under basic conditions occurs only from the syn adduct, thus forming 1-fluoro-3,4-dihydronaphthalene. Halofluorinations of 1,4-dihydronaphthalene also occur stereospecifically anti, and elimination under basic conditions gives naphthalene.

The mechanism of electrophilic addition has been widely investigated, both from the kinetic and stereochemical points of view.[1] Apart from the relative importance of the various kinetically significant processes, it is now known that the nature of the intermediates of the addition depends on the structure of the substrate, on the halogen and on the reaction medium, ranging from strongly bridged ions (type **C**), to weakly bridged species (type **B**) or open ions like **A**.



Scheme 1.

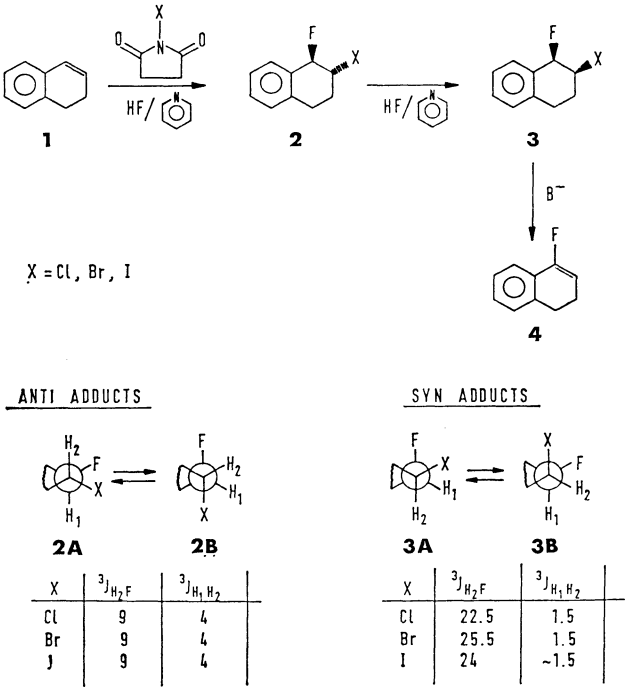
If the cation is of the open structure (**A**, X=F), a mixture of syn and anti adducts is generally expected. However, ion pairing phenomena can cause preferential formation of the syn adduct and electronic, steric or conformational effects can cause attack at one or other side of the carbonium p-orbital of **A** to be favoured. On the other hand, the intermediate can have a bridged structure (**C**, X=Br), which will presumably be opened to form an anti adduct. Available data on the addition of "BrX" species to olefins are sparse. In the steroid series,[2,3,4] stereospecific anti addition with Markovnikov type regioselectivity is observed; on the other hand, the bromofluorination of carbohydrates[5,6] is stereospecifically syn. Using hydrogen fluoride–pyridine in conjunction with *N*-bromosuccinimide for fluorination of aliphatic olefins, Olah and his coworkers[7] observed typical Markovnikov type regioselectivity. Bromofluorination of phenyl substituted olefins proceeds with Markovnikov type regioselectivity and the reaction is stereospecifically anti for trans and nonstereospecific for cis olefins.[8] The bromofluorination of 1-phenylcyclohexene also proceeds with Markovnikov type regioselectivity and is stereospecifically anti.[9] The halofluorinations of norbornene[10] and norbornadiene[11] with a mixture of *N*-halosuccinimide–hydrogen fluoride–pyridine have also been studied. Recently we found that fluorination of 1,2-dihydro- and 1,4-dihydronaphthalene occurs in different ways,[12] which stimulated us to investigate the reaction pathways involved in halofluorination of these systems.

Results and Discussion

The preparation of fluoroalkanes presents a different problem from that of other halogenoalkanes, and necessitates a specific method of fluorination.[13] Difficulties involve the handling of anhydrous hydrogen fluoride on the laboratory scale, the need for pressure equipment and low temperatures, and the ease of polymerisation of alkenes. Bromofluorination with hydrogen fluoride–pyridine–NBS avoids some experimental difficulties,[7] *e.g.* low temperature, high pressure techniques, and polymerisation of olefins.

Halofluorination of 1,2-Dihydronaphthalene. The 3 h reaction of 1,2-dihydronaphthalene (**1**), with a mixture of *N*-chlorosuccinimide–hydrogen fluoride–pyridine in ether at room temperature resulted in the formation of a crude reaction mixture, which showed two signals in its ^{19}F NMR spectrum: $\delta F = -158.5$ and -176 in the ratio 1.78:1.

Reduction of the reaction time from 3 to 1.5 h resulted in the formation of only one product, which showed one signal in its ^{19}F NMR spectrum at $\delta F =$



X	$^3J_{H_2F}$	$^3J_{H_1H_2}$
Cl	9	4
Br	9	4
I	9	4

X	$^3J_{H_2F}$	$^3J_{H_1H_2}$
Cl	22.5	1.5
Br	25.5	1.5
I	24	~1.5

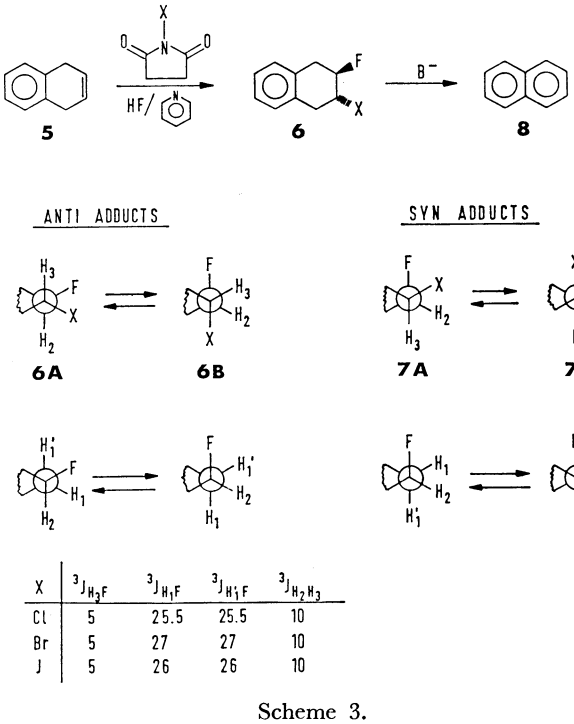
Scheme 2.

−158.5. Treatment of the reaction mixture formed in a 3 h halofluorination reaction with K–O–*t*-Bu in *t*-BuOH at T=50 °C resulted in the formation of a crude reaction mixture which showed a new signal at δF=−123.75, besides the signal at δF=−158.5, with the signal ratio of 1.7:1. Separation of the reaction mixture by TLC gave two products. The major product formed showed a doublet of multiplet signal at δF =−158.5 ($^2J_{FH}$=56 Hz), in its ^{19}F NMR spectrum and in its proton spectrum two signals at lower field: δH_1 =5.4, δH_2=4.6. The mass spectrum showed the following signals: *m/e*: 186 (M^++2, 20%), 184 (M^+, 60%), 148 (60), 129 (50), 122 (100), 115 (15). The second product showed a doublet signal at δF= −123.75, ^3J=15 Hz in its ^{19}F NMR, and in its mass spectrum the following signals: *m/e*: 148 (M^+, 63%), 147 (65), 146 (38), 133 (44). On the basis of the spectroscopic data we established that the crude reaction mixture formed under basic conditions contained 1-fluoro-2-chloro-1,2,3,4-tetrahydronaphthalene, and as the minor product, 1-fluoro-3,4-dihydronaphthalene (**4**). We were unable to separate the two products formed by the 3 h chlorofluorination reaction. However, on the basis of differences in their NMR spectra, which are shown in Scheme 2, and in the stability of the products under basic conditions, we have established that the major product formed (**2**) was *trans*-1-fluoro-2-chloro-1,2,3,4-tetrahydronaphthalene, while the second product, which was not formed in the case of the 1.5 h reaction, and which transformed under basic conditions to 1-fluoro-3,4-dihydronaphthalene, was found to be *cis*-1-fluoro-2-chloro-1,2,3,4-tetrahydronaphthalene (**3**).

A 3 h reaction of 1,2-dihydronaphthalene with a mixture of NBS–hydrogen fluoride–pyridine resulted in a crude reaction mixture, which showed two signals in its ^{19}F NMR spectrum: δF=−149 and −169 in the ratio 1.5:1, while reduction of the reaction time to 0.5 h resulted in the formation of only one product with the ^{19}F NMR signal at δF=−149. Under basic conditions, heating (T=50 °C) the mixture obtained in the 3 h bromofluorination reaction resulted in a mixture containing 1-fluoro-2-bromo-1,2,3,4-tetrahydronaphthalene and 1-fluoro-3,4-dihydronaphthalene (**4**) in the ratio 1.5:1. We were unable to separate the two products formed by the 3 h bromofluorination reaction. However, on the basis of differences in their NMR spectra, which are shown in Scheme 2, and in the stability of the products under basic conditions, we established that the major product formed (**2**) was *trans*-1-fluoro-2-bromo-1,2,3,4-tetrahydronaphthalene, while the second product, which was not formed in the case of a 0.5 h reaction and which was transformed under basic conditions to 1-fluoro-3,4-dihydronaphthalene, was found to be *cis*-1-fluoro-2-bromo-1,2,3,4-tetrahydronaphthalene (**3**).

Fifteen minutes iodofluorination with a mixture of *N*-iodosuccinimide–hydrogenfluoride–pyridine of 1,2-dihydronaphthalene resulted in the formation of a crude reaction mixture which showed two signals in its ^{19}F NMR spectrum, δF=−139 and −157, in the ratio 1.5:1. On the basis of a comparison (Scheme 2) of the NMR data of the products to those formed by chloro and bromofluorination, we established that the major product formed was *trans*-1-fluoro-2-iodo-1,2,3,4-tetrahydronaphthalene (**2**), and that the minor product formed was *cis*-1-fluoro-2-iodo-1,2,3,4-tetrahydronaphthalene (**3**). Reduction of the reaction time to 5 minutes resulted in the formation of product (**2**), while treatment of the reaction mixture obtained by 15 minutes iodofluorination under basic conditions resulted in a mixture containing products (**2**) and (**4**) in the ratio 1.5:1.

Halofluorination of 1,4-Dihydronaphthalene. A 5 h reaction of the mixture *N*-chlorosuccinimide–hydrogen fluoride–pyridine with 1,4-dihydronaphthalene at room temperature resulted in the formation of one product (**6**), which showed one signal in its ^{19}F NMR spectrum at δF=−177.5 (dtd, $^2J_{FH_2}$=51 Hz, $^3J_{FH_1}$=25.5 Hz, $^3J_{FH_3}$=5 Hz), and in its ^{1}H spectrum two signals at lower field. δH_2=4.7 (ddd, $^2J_{H_2F}$=51 Hz, $^3J_{H_2H_3}$= 10 Hz, $^3J_{H_1H_2}$=5 Hz), δH_3=4.3 (ddd, $^3J_{H_3F}$=5 Hz, $^3J_{H_3H_2}$=10 Hz, $^3J_{H_3H_4}$=5 Hz). In its mass spectrum the product showed the following signals: *m/e*: 186 (M^+ +2, 33%), 184 (M^+, 100%), 148 (53), 147 (43), 129 (85), 104 (45). 3 h heating (T=50 °C) of the adduct **6** resulted, under basic conditions, in the formation of naphthalene (**8**).



X	$^3J_{H_2F}$	$^3J_{H_1F}$	$^3J_{H_1'F}$	$^3J_{H_2H_3}$
Cl	5	25.5	25.5	10
Br	5	27	27	10
J	5	26	26	10

Scheme 3.

On the basis of the spectroscopic data and chemical transformation, we established that 2-fluoro-3-chloro-1,2,3,4-tetrahydronaphthalene was formed. The Newman projection formulae of two possible isomers are shown in Scheme 3. On the basis of coupling constants we have established that the trans adduct was formed and that conformation **6A** is the favoured one.

A 3 h reaction of the mixture *N*-bromosuccinimide–hydrogenfluoride–pyridine with 1,4-dihydronaphthalene resulted in the formation of one product (**6**), which

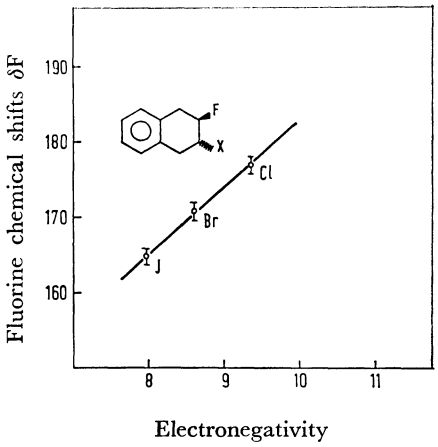


Fig. 1. Dependence of δF on electronegativity of the halogen atom.

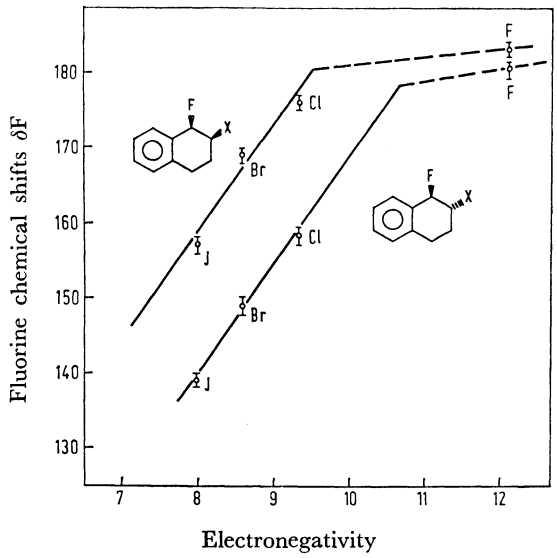


Fig. 2. Dependence of δF on electronegativity of the halogen atom.

showed one signal in its ^{19}F NMR at δF=−171 (dtd, $^{2}J_{FH_2}$=51 Hz, $^{3}J_{FH_1}$=27 Hz, $^{3}J_{FH_3}$=5 Hz) and in its proton spectrum two signals at lower field: δH_2=4.9 (ddd, $^{2}J_{H_2F}$=51 Hz, $^{3}J_{H_2H_1}$=5 Hz, $^{3}J_{H_2H_3}$=10 Hz), δH_3=4.3 (ddd, $^{3}J_{H_3F}$=5 Hz, $^{3}J_{H_3H_2}$=10 Hz, $^{3}J_{H_3H_4}$=5 Hz). In its mass spectrum, product **6** showed the following signals: *m/e*: 230 (M^++2, 60%), 228 (M^+, 60), 129 (100), 128 (50), 104 (16). Adduct **6** was also converted under basic conditions to naphthalene (**8**). On the basis of the spectroscopic data and the chemical transformation we established that *trans*-2-fluoro-3-bromo-1,2,3,4-tetrahydronaphthalene was formed. In this case also the preferential conformation is **6A**.

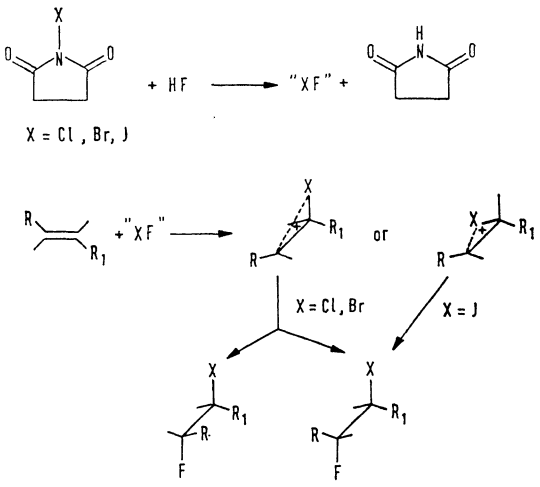
A 15-minute reaction of the mixture *N*-iodosuccinimide–hydrogenfluoride–pyridine with 1,4-dihydronaphthalene resulted in the formation of one product which showed one signal in its ^{19}F NMR spectrum at δF=−165 (dtd, $^{2}J_{FH_2}$=52 Hz, $^{3}J_{FH_1}$=26 Hz, $^{3}J_{FH_3}$=5 Hz), and in its proton spectrum two signals at lower field δH_2=5 (ddd, $^{2}J_{H_2F}$=52 Hz, $^{3}J_{H_2H_3}$=10 Hz, $^{3}J_{H_2H_1}$=5 Hz), δH_3=4.5 (ddd, $^{3}J_{H_3F}$=5 Hz, $^{3}J_{H_3H_2}$=10 Hz, $^{3}J_{H_3H_4}$=5 Hz). In its mass spectrum the product **6** showed the following signals: *m/e*: 276 (M^+, 45%), 149 (80), 130 (33), 129 (85), 128 (100), 127 (35), 115 (23). Product **6** was also converted under basic conditions to naphthalene. On the basis of the spectroscopic data and the chemical transformation, we established that *trans*-2-fluoro-3-iodo-1,2,3,4-tetrahydronaphthalene was formed. Again the preferential conformation was found to be **6A**.

The dependence of fluorine chemical shifts in *trans*-2-fluoro-3-halo-1,2,3,4-tetrahydronaphthalene on the Mulliken electronegativity of the halogen atom is presented in Fig. 1, while in Fig. 2 the influence of Mulliken electronegativity on chemical shifts of the fluorine atom in cis and trans 1-fluoro-2-halo-1,2,3,4-tetrahydronaphthalene is shown.

The correlation is good in the case of *trans*-2-fluoro-3-halo-1,2,3,4-tetrahydronaphthalene and *trans*-2-fluoro-3-halo-1,2,3,4-tetrahydronaphthalene, while in the case of cis adducts the correlation is worse. From Figs. 1 and 2 it can also be seen that the influence of different electronegativities of the halogen atom on fluorine chemical shifts is greater in the case of *trans*-1-fluoro-2-halo-1,2,3,4-tetrahydronaphthalene ($\Delta\delta$F/Δelectronegativity=13.4) than in the case of *trans*-2-fluoro-3-halo-1,2,3,4-tetrahydronaphthalene ($\Delta\delta$F/Δelectronegativity =9.4). Introduction of the values of fluorine chemical shifts[12] for cis and trans 1,2-difluoro-1,2,3,4-tetrahydronaphthalene into Figure 2 shows that these values strongly deviate from the straight line connecting the values for chloro, bromo and iodo derivatives, which could be ascribed to the different conformation of the difluoride and, or, through space interaction between fluorine atoms.

For bromofluorination with *N*-bromosuccinimide in the presence of hydrogen fluoride, reaction sequences involving a cyclic bromonium ion have been suggested.[3,4,14] In general, one can suggest a reaction between *N*-halosuccinimide and hydrogen fluoride, resulting "XF" species which can then react with the double bond, following Markovnikov type regioselectivity.

However, it is known that β-chlorocarbonium ions have only partly bridged structures, while the strongly

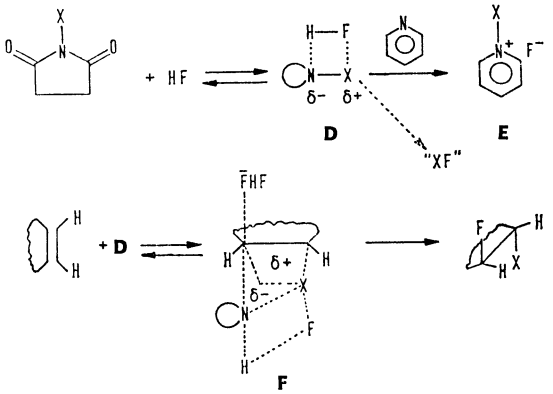


Scheme 4.

bridged halonium ions can be expected only in the case of iodofluorination with "IF" species. So, in the case of chlorofluorination and bromofluorination with "XF" species, syn and anti adducts should be observed, while in the case of iodofluorination only an anti-adduct is to be expected. We suggest a reaction pathway (Scheme 5), which can better explain the anti stereoselectivity. We propose the formation of a polarized *N*-halosuccinimide–hydrogen fluoride complex (**D**), which reacts in a reversible step with 1,2-dihydro- or 1,4-dihydronaphthalene to form a complex (**F**), decomposing in the next step to halofluorides. The reversible step, leading to complex (**F**), could explain the high degree of isomerization of cis alkenes.[8] The formation of "XF" species could be involved only in iodofluorination with the mixture of *N*-iodosuccinimide–hydrogen fluoride–pyridine.

A complex similar to (**F**) was suggested for bromination of 1-phenylcyclohexene with bromine–pyridine complex[15] and has also been proposed for E2Hal elimination of bromine from *trans*-1,2-dibromocyclohexane with benzenethiolate as base.[16] A polarized complex of NBS with olefins has been suggested in the bromination of styrene and cyclohexene with NBS in the presence of dimethylsulfoxide and methanol.[17]

A third interpretation presents the halofluorination reaction as involving the formation of the pyridinium–halide complex (**E**), reacting with 1,2- or 1,4-dihydronaphthalene with high stereoselectivity. *trans*-1-Fluoro-2-halo-1,2,3,4-tetrahydronaphthalenes, the only products formed in shorter reaction times, are unstable under the reaction conditions and isomerize to cis adducts.



Scheme 5.

Experimental

Procedure. IR spectra were recorded with a Perkin-Elmer 257 spectrometer, ^1H and ^{19}F NMR spectra with a JEOL JNM-PS-100 (CCl_4 or $CDCl_3$ as solvent and Me_4Si or CCl_3F as internal reference). Mass spectra, including high resolution measurements, were carried out on a CEC-21-110 spectrometer. GLPC was carried out with a Varian Aerograph 1800 instrument and TLC with Merck chromatoplates (PSC-Fertig-platten silica gel F-254, activated for 2 h at 120 °C before use).

Materials. A solution of pyridine–hydrogen fluoride was prepared according to Olah's procedure.[7] The pyridine used was predistilled, while hydrogen fluoride (Fluka, Purum) was used without prior purification. *N*-halosuccinimides (Fluka, Purum) were crystallised and dried (P_2O_5) before use. Diethyl ether was purified by standard methods and distilled before use. 1,2- and 1,4-dihydronaphthalene were prepared by known methods from naphthalene[18] and purified before use.

Addition and Isolation Procedures. In a mixture of hydrogen fluoride in pyridine (70% solution, 2 ml) and ether (2 ml), *N*-halosuccinimide (1.2 mmol) was dissolved with stirring at 0 °C for 10 min, and then the olefin (1 mmol) was added. The mixture was left at room temperature for various reaction times with stirring, then poured into ice-water and extracted with ether. The ether layer was washed with water, aqueous sodium hydrogencarbonate, then water again, dried (Na_2SO_4) and evaporated. The NMR spectra of crude reaction mixtures were recorded and then the pure products were obtained by preparative TLC or by crystallisation from the corresponding solvent. The structure of pure products were established from ^{19}F and ^1H NMR mass and IR spectral data.

trans-*1-Fluoro-2-chloro-1,2,3,4-tetrahydronaphthalene (**2a**)*:
2a is an oily product (48%) (purified by preparative TLC, SiO_2; cyclohexane:chloroform:triethylamine 95:4.5:0.5). NMR: $\delta F = -158.5$ (dm), $\delta H_1 = 5.40$ (dd), $\delta H_2 = 4.60$ (m), δCH_2-$CH_2 = 2$ to 3 (m), δPh (m) $= 7.30$ $^2J_{FH_1} = 56$ Hz, $^3J_{FH_2} = 9$ Hz, $^3J_{H_1H_2} = 4$ Hz. MS: calcd for $C_{10}H_{10}ClF$ *m/e* 184.0449, found 184.0448 *m/e*: 186 ($M^+ + 2$, 20%), 184 (M^+, 60), 149 (20), 148 (60), 147 (20), 129 (50), 128 (35), 122 (100), 115 (15), 104 (10).

trans-*1-Fluoro-2-bromo-1,2,3,4-tetrahydronaphthalene (**2b**)*:
White crystals (52%), mp=29—30 °C, (purified by preparative TLC under conditions mentioned below). NMR: $\delta F = -149$ (dm), $\delta H_1 = 5.60$ (dd), $\delta H_2 = 4.60$ (m), δCH_2—$CH_2 = 2.2$ to 2.8 (m), $\delta Ph = 7.30$ (m), $^2J_{FH_1} = 54$ Hz, $^3J_{FH_2} = 9$ Hz, $^3J_{H_1H_2} = 4$ Hz. MS: calcd for $C_{10}H_{10}BrF$ *m/e* 227.9964, found 227.9966, *m/e* 230 ($M^+ + 2$; 35%), 228 (M^+, 35%), 210 (16), 208 (16), 149 (40), 148 (48), 147 (25), 146 (15), 129 (100), 128 (60), 127 (24), 115 (21), 104 (10), 102 (10).

trans-*1-Fluoro-2-iodo-1,2,3,4-tetrahydronaphthalene (**2c**)*:
White crystals (75%) (from ethanol) decomposing at room temperature. NMR: $\delta F = -139$ (dm), $\delta H_1 = 5.60$ (dd), $\delta H_2 = 4.60$ (m), δCH_2—$CH_2 = 2.2$ to 2.8 (m), $^2J_{FH_1} = 54$ Hz, $^3J_{FH_2} = 9$ Hz, $^3J_{H_1H_2} = 4.5$ Hz. MS: calcd for $C_{10}H_{10}FJ$ *m/e* 275.9813, found 275.9816 *m/e* 276 (M^+, 28%), 149 (100), 129 (75), 128 (81), 127 (25), 115 (20), 102 (11).

trans-*2-Fluoro-3-chloro-1,2,3,4-tetrahydronaphthalene (**6a**)*:
White crystals (60%), mp=29—30 °C, (purified by preparative TLC), NMR: $\delta F = -177.5$ (dtd), $\delta H_2 = 4.9$ (ddd), $\delta H_3 = 4.3$ (ddd), $\delta H_1 = 2.9$ (m), $\delta H_4 = 3.7$ (m), $\delta Ph = 7.1$ (m), $^2J_{FH_2} = 51$ Hz, $^3J_{FH_3} = 5$ Hz, $^3J_{FH_1} = 25.5$ Hz, $^3J_{FH_1'} = 25.5$ Hz, $^3J_{H_2H_3} = 10$ Hz, $^3J_{H_1H_2} = 5$ Hz, $^3J_{H_3H_4} = 5$ Hz. MS: calcd for $C_{10}H_{10}ClF$ *m/e* 184.0449, found 184.0449, *m/e*: 186 ($M^+ + 2$; 33%), 184 (M^+, 100), 149 (34), 148 (54), 147 (43), 146 (21), 129 (85), 128 (43), 127 (16), 115 (18), 104 (45), 103 (10).

trans-*2-Fluoro-3-bromo-1,2,3,4-tetrahydronaphthalene (**6b**)*:
White crystals (68%), mp=35—37 °C, (purified by preparative TLC), NMR: $\delta F = -171$ (dtd), δ $H_2 = 4.9$ (ddd), δ $H_3 = 4.3$ (ddd), δ $H_1 = 2.9$ (m), δ $H_4 = 3.7$ (m), δ Ph=7.1, $^2J_{FH_2} = 54$ Hz, $^3J_{FH_3} = 5$ Hz, $^3J_{FH_1} = 27.0$ Hz, $^3J_{FH_1'} = 27.0$ Hz, $^3J_{H_2H_3} = 10$ Hz, $^3J_{H_1H_2} = 5$ Hz, $^3J_{H_3H_4} = 5$ Hz. MS: calcd for $C_{10}H_{10}BrF$ *m/e* 227.9964, found 227.9940, *m/e*: 230 ($M^+ + 2$; 60%), 228 (M^+, 60), 149 (31), 148 (35), 147 (28), 146 (17), 129 (100), 128 (50), 115 (20), 104 (16).

trans-*2-Fluoro-3-iodo-1,2,3,4-tetrahydronaphthalene (**6c**)*:
White crystals (70%) (from ethanol), mp=57—58 °C.

NMR: $\delta F = -165$ (dtd), $\delta H_2 = 5.0$ (ddd), $\delta H_3 = 4.5$ (ddd), $\delta H_1 = 3.2$, $\delta H_4 = 3.7$ (m), $\delta Ph = 7.1$, $^2J_{FH_2} = 52$ Hz, $J_{FH_3} = 5$ Hz, $^3J_{FH_1} = 26$ Hz, $^3J_{FH_1'} = 26$ Hz, $^3J_{H_2H_3} = 10$ Hz, $^3J_{H_1H_2} = 5$ Hz, $^3J_{H_3H_4} = 5$ Hz. MS: calcd for $C_{10}H_{10}FJ$ *m/e* 275.9813, found 275.9825, *m/e*: 276 (M^+, 45%), 149 (80), 148 (7), 147 (14), 146 (14), 129 (85), 128 (100), 127 (35), 126 (13), 102 (20).

Treatment of the Mixture of trans- *and* cis-*1-Fluoro-2-halo-1,2,3,4-tetrahydronaphthalene with Base.* To a crude reaction mixture obtained by halofluorination of 1,2-dihydronaphthalene (longer reaction time), a solution of 1.3 mmol of potassium *t*-butoxide in *t*-butylalcohol (2 ml) was added, and the mixture heated for 3—4 h at $T = 50$ °C, then poured into water (10 ml), exacted with ether, the ether layer washed with water (5 ml twice), dried and evaporated. NMR spectra of the crude reaction mixture were recorded to obtain the product distribution, and then it was separated by preparative TLC (SiO_2, cyclohexane: chloroform: triethylamine 95:4.5:0.5). Products (**2**), and 1-fluoro-3,4-dihydronaphthalene (**4**) were isolated. Spectroscopic data for **4** were in agreement with those already published.[12]

Treatment of trans-*2-Fluoro-3-halo-1,2,3,4-tetrahydronaphthalene with Base.* To a solution of 1.5 mmol of potassium *t*-butoxide in *t*-butyl alcohol (2 ml), 1 mmol of product **6** was added and the mixture heated for 3—4 hours at 50 °C. The reaction mixture was poured into water (10 ml), extracted with ether, the ether layer washed with water (5 ml, twice), dried (Na_2SO_4) and evaporated. NMR spectra of the crude reaction mixture were recorded, showing that naphthalene (**8**) was the only product formed.

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