(Fluoroorgano)fluoroboranes and -borates. 7 [1]

The Reaction of R_FBF_2 and K $[R_FBF_3]$ (R_F = perfluorophenyl-, perfluoroalk-1-enyl- and perfluoroalkyl) with Xenon Difluoride in Anhydrous HF

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Abstract. The dissolution of (perfluoroorgano)difluoroboranes R_FBF_2 in anhydrous HF (aHF) resulted in equilibrium mixtures of the starting borane and different kinds of acid-base products: $[H_2F]$ [$R_FBF_2(F \cdot HF)$] ($R_F = C_6F_5$, *cis*- $C_2F_5CF=CF$, *trans*- $C_4F_9CF=CF$) or [H_2F] [R_FBF_3] ($R_F = C_6F_{13}$). In aHF the aryl compounds $C_6F_5BF_2$ and K [$C_6F_5BF_3$] showed two parallel reactivities with XeF_2: xenodeborylation (formation of the [C_6F_5Xe]⁺ cation) and fluorine addition to the aryl group. In aHF perfluoroalk-1-enyldi-

fluoroboranes R_FBF_2 as well as potassium perfluoroalk-1-enyltrifluoroborates K [R_FBF_3] ($R_F = cis$ -C₂F₅CF=CF, *trans*-C₄F₉CF= CF) underwent only fluorine addition across the carbon-carbon double bond under the action of XeF₂. Potassium perfluorohexyltrifluoroborate K [C₆F₁₃BF₃] did not react with XeF₂ in aHF.

Keywords: Fluoroboranes; Fluoroborates; Fluorine addition; Xenodeborylation; NMR spectroscopy

(Fluororgano)fluorborane und -fluoroborate. 7 [1]

Die Reaktion von R_FBF_2 und K $[R_FBF_3]$ (R_F = Perfluorphenyl-, Perfluoralk-1-enylund Perfluoralkyl) mit Xenondifluorid in wasserfreiem Fluorwasserstoff

Inhaltsübersicht. Beim Lösen von (Perfluororgano)difluorboranen R_FBF_2 in wasserfreier HF (aHF) stellten sich Gleichgewichtsgemische zwischen den Ausgangsboranen und unterschiedlichen Arten von Säure-Base Produkten ein: [H₂F] [$R_FBF_2(F \cdot HF)$] ($R_F = C_6F_5$, *cis*- $C_2F_5CF=CF$, *trans*- $C_4F_9CF=CF$) oder [H₂F] [R_FBF_3] ($R_F = C_6F_{13}$). Die Arylverbindungen $C_6F_5BF_2$ und K [$C_6F_5BF_3$] wiesen in ihren Umsetzungen mit XeF₂ in aHF zwei parallele Re-

aktivitäten auf: die Xenodeborylierung (Bildung des $[C_6F_5Xe]^+$ Kations) und die Fluoraddition an die Arylgruppe. Sowohl bei den Perfluoroalk-1-enyldifluorboranen R_FBF_2 als auch bei den Kaliumperfluoralk-1-enyltrifluoroboraten K $[R_FBF_3]$ ($R_F = cis$ - $C_2F_5CF=CF$, *trans*- $C_4F_9CF=CF$) erfolgte mit XeF₂ in aHF nur Fluoraddition an die C-C Doppelbindung. Kaliumperfluorhexyltrifluoroborat K $[C_6F_{13}BF_3]$ reagierte nicht mit XeF₂ in aHF.

Introduction

Recent studies of the reaction of potassium organotrifluoroborates K [RBF₃] with protic acids of different strength showed that the salts with R = alkyl, alk-1-enyl, phenyl and the fluorine-containing alk-1-enyl group R'CF=CF (R' = F, Cl, C₃F₇O, *trans*-C₄H₉) underwent hydrodeboration when treated with CH₃CO₂H, CF₃CO₂H or aqueous HF at 20 - 50 °C. When R was a perfluorinated alkyl, alk-1-enyl or phenyl group, the salts K [RBF₃] did not react even with the strong acid, aHF, at room temperature [1]. A similar

Results and Discussion

Solvolysis of (perfluoroorgano) difluoroboranes in aHF

In contrast to boron trifluoride, perfluorohexyldifluoroborane **1**, perfluoro-*trans*-hex-1-enyldifluoroborane **2**, perfluoro-*cis*-but-1-enyldifluoroborane **3** and pentafluorophenyldifluoroborane **4** were well soluble in aHF at room temperature, but below -20 to -30 °C their solubility was

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resistance towards the hydrodeboration in aHF was observed for perfluoro-*trans*-hex-1-enyldifluoroborane [2]. The inertness of these perfluorinated trifluoroborates in aHF prompted us to extend our previous research into the xenodeborylation of tris(pentafluorophenyl)borane and the borate Cs[(C₆F₅)₃BF] with xenon difluoride in aHF [3, 4], and to investigate some prototypes of perfluorinated organotrifluoroborates and -difluoroboranes in the reaction with XeF₂ in aHF.

significantly decreased and white suspensions were formed.



As strong Lewis acids, the boranes $R_FBF_2 \mathbf{1} - \mathbf{4}$ interacted with the fluoride donor aHF to a different extent. For instance, the ¹⁹F NMR spectrum of the alkyldifluoroborane 1 in aHF (-20 °C) contained a BF_x resonance at -150.60 ppm with a relative intensity equal to that of the trifluoromethyl group ($\delta = -80.71$) of the perfluorinated hexyl chain. -150.60 ppm is a characteristic position of BF_3 groups [5]. However, the ¹⁹F NMR spectra of the less acidic alkenyl and aryl difluoroboranes 2 - 4 showed no resonances of the boron-bonded fluorine atoms whereas the ¹⁹F NMR spectra of CF₂=CFBF₂ and ClCF=CFBF₂ in aHF (-40 °C) did not differ from the spectra in CH₂Cl₂ [2]. This means that the less acidic alkenylboranes R_FBF_2 $(R_F = CF_2 = CF, CCIF = CF)$ did not abstract the fluoride ion from aHF, while the more acidic boranes ($R_F = cis$ -C₂F₅, trans-C₄F₉, C₆F₅) partially abstracted fluoride. In the case of the most acidic alkylborane 1 the equilibrium was practically completely shifted to the trifluoroborate anion (eqs. (1a) and (1b)).

In order to understand the reactivity of R_FBF_2 towards XeF_2 it is important to emphasize two main aspects of the system R_FBF_2/aHF : (a) the significant increase in acidity of the media after dissolution of the boranes R_FBF_2 in aHF ("acidified" HF) and (b) the presence of the trifluoroborate anions $[R_FBF_3]^-$ or the donor-acceptor complexes $[R_FBF_2(F \cdot HF)]^-$ in these solutions. Both aspects play a significant role in the reactions with xenon difluoride. Indeed, the high Brønsted acidity of the media increases the contribution of the polarized form of XeF_2 (borderline formulation: $[FXe]^+$) which is a very strong oxidant $\{EA([FXe]^+) = 10.6 - 10.9 \text{ eV} [6, 7]\}$.

Furthermore, the products of the solvolysis of R_FBF_2 - $[R_FBF_3]^-$ or $[R_FBF_2(F \cdot HF)]^-$ are more electron-rich species than the parent organodifluoroboranes and can be more readily oxidized. Consequently, the oxidation (addition of fluorine to C=C double bonds) should be expected as a competitive reaction route to xenodeborylation when the unsaturated perfluoroorganodifluoroboranes are allowed to react with xenon difluoride in aHF.

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Reactions of R_FBF_2 with XeF_2 in aHF ("acidified" HF)

Previously we have shown that the reaction of pentafluorophenyldifluoroborane with xenon difluoride in CH_2Cl_2 gave pentafluorophenylxenon(II) tetrafluoroborate in high yield [8]. However, in aHF the formation of a mixture of perfluorinated phenyl- and cyclohexa-1,4-dienylxenonium cations, cyclohexa-1,4-dienyltrifluoroborate and tetrafluoroborate anions was found after the quantitative conversion of the reactants (¹⁹F NMR). The observed excess of anions over cations can be explained by the presence of $[H_2F]^+$ (eq. (1b). Fast fluoride exchange did not allow to detect the ¹⁹F NMR signal of $[H_2F]^+$.



The same products were recently obtained by the reaction of tris(pentafluorophenyl)borane with XeF₂ in aHF, but a direct quantitative comparison is difficult because of the heterogeneous nature of that reaction [3]. But in both cases the pentafluorophenyl-containing boranes $C_6F_5BX_2$ (X = F, C_6F_5) underwent both xenodeborylation and fluorine addition to the pentafluorophenyl ring.

In contrast, no organoxenon compounds were formed in the reaction of xenon difluoride with the alkenylboranes 2 and 3. Essentially fluorine addition across the carbon-carbon double bond took place at -30 to -20 °C.



Reactions of $K [R_F B F_3]$ with $X e F_2$ in aHF ("basic" HF)

The dissolution of organotrifluoroborates in aHF results primarily in the protonation of the fluorine atoms bonded to boron. This leads to a fluoroborate/fluoroborane equilibrium and a decrease in the acidity of HF. The transformation of the organotrifluoroborate into the corresponding organodifluoroborane is accompanied by the reduction of the coordination number at boron [1] and was expected to favour the xenodeborylation reaction relative to the oxidative addition of fluorine. Indeed, the reaction of potassium pentafluorophenyltrifluoroborate **5** with xenon difluoride in aHF gave predominantly pentafluorophenylxenon(II) tetrafluoroborate. The oxidative fluorination of the $[C_6F_5BF_3]^-$ anion occurred to a minor extent.



It should be noted that the treatment of $Cs[(C_6F_5)_3BF]$ with XeF₂ (3 equiv.) in aHF (20 °C, 15 minutes) yielded a similar ratio of xenodeborylation to oxidative fluorine addition [3].

However, a decrease of the acidity of the media did not influence the reaction route of potassium perfluoroalk-1-enyltrifluoroborates with xenon difluoride. Salts 6, 7 and 8 were quickly converted at -30 °C into the potassium perfluoroalkyltrifluoroborates 9, 10 and 11, respectively. The additional presence of KF (1 equiv.) reduced the reaction rate of the borate 7 with XeF₂ but had no effect on the course of the reaction.



$$\begin{array}{cccc} & & & \mathsf{KF}, \mathsf{aHF} \\ [\mathit{cis}\text{-}\mathsf{C}_2\mathsf{F}_5\mathsf{C}\mathsf{F}\text{=}\mathsf{C}\mathsf{F}\mathsf{B}\mathsf{F}_3] + \mathsf{Xe}\mathsf{F}_2 & & & & \\ \hline \mathbf{7} & & & & \mathsf{20}\,^\circ\mathsf{C}, \, \mathsf{1.5}\,\mathsf{h} & & \mathbf{10}\,(\mathsf{93}\,\%) \end{array}$$

Potassium perfluorohexyltrifluoroborate 11 did not react with XeF_2 in aHF at room temperature within 1.5 hours.

Within the framework of the proposed mechanism of the xenodeborylation [4] the inertness of potassium perfluoroalkyltrifluoroborates towards xenon difluoride in aHF is explained by the negligible equilibrium concentration of perfluoroalkyldifluoroborane which is necessary for the realization of the F-Xe \cdot F \cdot F₂BR transition state [4]. This is in agreement with the earlier explanation of the inertness of [Me₄N] [(C₆F₅)₄B] towards XeF₂ in aHF solution [3].

The oxidative addition of fluorine with XeF_2 in aHF to the C=C bond of perfluoroalk-1-enyldifluoroboranes as well as potassium perfluoroalk-1-enyltrifluoroborates occurred for two essential reasons. First, the highly acidic media cause a dramatic increase of the oxidation potential of xenon difluoride. The role of the [FXe]⁺ cation or a related polarized form of XeF₂ in the oxidative fluorination of (C₆F₅)₃B in aHF was discussed in [3]. This aspect was underlined by the model reaction of XeF₂ with hexafluorobenzene. C₆F₆ did not react with XeF₂ in aHF in the presence of K [HF₂] ("basic" HF) while in neat anhydrous hydrogen fluoride the fluorine addition proceeded within a few minutes.



The second reason is the higher reducing potential of fluoroalkenyl or aryl groups in organotrifluoroborate anions, compared to that of the corresponding organodifluoroboranes. Both aspects are important for the reaction of perfluoroalk-1-enyldifluoroboranes with XeF_2 in aHF and explain the fast oxidative addition of fluorine.

Experimental

NMR spectra were recorded on Bruker spectrometers, WP 80 SY (¹⁹F at 75.39 MHz), AVANCE 300 (¹¹B at 96.29 MHz, ¹⁹F at 282.40 MHz). The chemical shifts are referenced to BF₃ · OEt₂/CDCl₃ 15 % v/v (¹¹B) and CFCl₃ (¹⁹F) (C₆F₆ as a secondary reference, $\delta = -162.9$). Superscripts with the number of the related carbon atom are used for the designation of the fluorine atoms of fluoroorganic chains. The composition of the reaction mixtures and the yields of products were determined by ¹⁹F NMR spectroscopy. The IR spectra were recorded on a Bruker Vector 22 FT-spectrometer on KBr pellets. The elemental analysis was performed in the N. N. Vorozhtsov Novosibirsk Institute of Organic Chemistry (Novosibirsk, Russia).

Hydrogen fluoride was dried by electrolysis (stainless steel cell, Nielectrodes). All manipulations with anhydrous HF were performed in FEP (block copolymer of tetrafluoroethylene and hexafluoropropylene) equipment.

The potassium (perfluoroorgano)trifluoroborates 5 [9], 6 - 8 [10], 11 [11] and (perfluoroorgano)difluoroboranes 1, 2, 3, 4 [2, 9, 11] were prepared using literature procedures.

Solvolysis of (perfluoroorgano) difluoroboranes in aHF.

The solution of $C_6F_5BF_2$ (0.32 mmol) in CH_2Cl_2 (1.5 ml) was stirred with aHF (1 ml) at -20 °C. The upper acidic phase was decanted into a pre-cooled FEP trap. The solutions of *cis*- $C_2F_5CF=CFBF_2$ (0.42 mmol), *trans*- $C_4F_9CF=CFBF_2$ (0.22 mmol) and $C_6F_{13}BF_2$ (0.12 mmol) in aHF (0.5 - 1 ml) were prepared in a similar manner. After recording the ¹⁹F NMR spectra these solutions were used for the following reactions with xenon difluoride.

 $C_6F_5BF_2$. ¹⁹F NMR (aHF, -20 °C): $\delta = -131.88$ (2F, *o*-F), -149.54 (t, ³*J*(*p*-F,*m*-F) 18 Hz, 1F, *p*-F), -162.05 (2F,*m*-F); the resonance of the boron-bonded fluorine atoms was not observed.

 $cis\text{-}C_2F_5CF\text{=}CFBF_2.$ ^{19}F NMR (aHF, $-20~^\circ\text{C}):$ $\delta=-83.18$ (3F, F⁴), -118.43 (2F, F³), -126.15 (1F, F²), -145.64 (1F, F¹); the resonance of the boron-bonded fluorine atoms was not observed.

trans-C₄F₉CF=CFBF₂. ¹⁹F NMR (aHF, -20 °C): $\delta = -79.53$ (3F, F⁶), -115.83 (2F, F³), -122.83 and -124.50 (2 CF₂), -159.83 (d, 1F, F¹), -169.17 (d, ³*J*(F²,F¹) 132 Hz, 1F, F²); the resonance of boron-bonded fluorine atoms was not observed (see also [2]).

 $C_6F_{13}BF_2. \, ^{19}F$ NMR (aHF, -20 °C): $\delta=-80.71$ (3F, $F^6),\,-121.64$ (2 CF₂), -122.62 (CF₂), -125.57 (2F, $F^5),\,-133.54$ (2F, $F^1),\,-150.60$ (3F, unresolved signal, BF_3).

Reactions of (perfluoroorgano) difluoroboranes with xenon difluoride in aHF

Solid xenon difluoride (total amount 0.61 mmol) was added in small portions at -30 °C to the stirred solution of *cis*-C₂F₅CF= CFBF₂ in aHF (see above). Evolution of xenon took place immediately. After addition of K [HF₂] in excess the solution was diluted with water, neutralized with K₂CO₃, and extracted with MeCN. The ¹⁹F NMR spectrum showed the presence of K [C₄F₉BF₃] (70 -75% yield), unreacted K [*cis*-C₂F₅CF=CFBF₃] (5 -7%) and an unknown product (15 -20%).

Similar treatment of the solution of *trans*- $C_4F_9CF=CFBF_2$ in aHF with solid XeF₂ (1 equiv.) at -20 °C led to the quantitative formation of [H₂F] [$C_6F_{13}BF_3$] (¹⁹F NMR).

The reaction of $C_6F_5BF_2$ (0.17 mmol) in aHF (1 ml) with solid XeF₂ (0.17 mmol) at -30 °C caused the immediate evolution of xenon and after 5 - 10 minutes the ¹⁹F NMR spectrum displayed the resonances of $[C_6F_5Xe]^+$, $[1,4-C_6F_7Xe]^+$, $[1,4-C_6F_7BF_3]^-$ and $[BF_4]^-$ (molar ratio 6 : 2 : 10 : 13), besides the resonance of aHF (-190 ppm). No signals of $C_6F_5BF_2$ and XeF₂ were still observed.

Reaction of potassium (perfluoroorgano)trifluoroborates with xenon difluoride in aHF

K [C₆F₅BF₃] (168 mg, 0.61 mmol) was dissolved in aHF (1.5 ml) at $-30 \,^{\circ}$ C and the cold (0 $^{\circ}$ C) solution of XeF₂ (119 mg, 0.70 mmol) in aHF (0.5 ml) was added. Gas evolution occurred and a yellow solution was formed. The reaction mixture was stirred at $-30 \,^{\circ}$ C for 10 min. and then 15 min. at 20 $^{\circ}$ C. The ¹⁹F NMR spectrum (0 $^{\circ}$ C) showed the absence of XeF₂ and K [C₆F₅BF₃] and the formation of [C₆F₅Xe]⁺, [BF₄]⁻, [1,4-C₆F₇BF₃]⁻ (molar ratio 15 : 15 : 6) and C₆F₆ (traces), besides the resonance of aHF (-190 ppm).

K [CF₂=CFBF₃] (140 mg, 0.74 mmol) was suspended in aHF (2 ml) at -60 °C and solid XeF₂ (132 mg, 0.78 mmol) was added with stirring. Slow warming to -30 °C was accompanied by the evolution of gas which was completed within 20 min. Evaporation of aHF in vacuum at 20 °C gave K [C₂F₅BF₃] (140 mg, 84 % yield). C₂BF₈K (225,92): calculated C 10.63, F 67.28; found C 10.5, F 66.6 %.

¹⁹F NMR (CH₃CN): δ = -83.08 (q, ⁴*J*(F²,B*F*₃) 4.8 Hz, 3F, F²), -135.92 (q, ²*J*(F¹,B) 19.6 Hz, 2F, F¹), -152.91 (q, ¹*J*(F,B) 41 Hz, 3F, B*F*₃). ¹¹B NMR (CH₃CN): δ = -0.74 (qt, ¹*J*(B,F) 41 Hz, ²*J*(B,F¹) 20 Hz).

Similarly, the reaction of K [*trans*-C₄F₉CF=CFBF₃] (143 mg, 0.36 mmol) with XeF₂ (72 mg, 0.42 mmol) in aHF (2 ml) at -30 °C resulted in K [C₆F₁₃BF₃] which was isolated in 95 % yield (145 mg). The reaction of K [*cis*-C₂F₅CF=CFBF₃] (120 mg, 0.41 mmol) with XeF₂ (83 mg, 0.49 mmol) in aHF (2 ml) at -30 °C yielded K [C₄F₉BF₃] (116 mg, 87 %).

K [*cis*-C₂F₅CF=CFBF₃] (143 mg, 0.49 mmol) and KF (42 mg, 0.53 mmol) were dissolved in aHF (1.5 ml) at -30 °C and the cold (0 °C) solution of XeF₂ (93 mg, 0.55 mmol) in aHF (0.5 ml) was added with stirring. After 15 min. the ¹⁹F NMR spectrum (-30 °C) revealed the resonances of the anions [*cis*-C₂F₅CF=CFBF₃]⁻ [δ = -82.81 (3F, F⁴), -119.14 (2F, F³), -139.57 (4F, F¹ and BF₃), -148.00 (1F, F²)], [C₄F₉BF₃]⁻ [δ = -79.43 (3F, F⁴), -122.34 (2F, F²), -124.36 (2F, F³), -132.45 (2F, F¹), -148.47 (3F, BF₃)], and XeF₂ (-195.90 ppm) (molar ratio 1 : 1 : 1.2). The solution was stirred at 20 °C for 1.5 h until the fluorine addition was completed

 $(^{19}F$ NMR) and after work up K $[C_4F_9BF_3]$ (149 mg, 93 % yield) was isolated.

C₄BF₁₂K (325,93): calculated C 14.74, F 69.95; found C 14.7, F 69.4 %.

¹⁹F NMR (CD₃CN): δ = -80.37 (tt, ⁴*J*(F⁴,F²) 9.9 Hz, ³*J*(F⁴,F³) 3.8 Hz, 3F, F⁴), -123.60 (m, 2F, F²), -125.38 (m, 2F, F³), -132.90 (m, 2F, F¹), -151.96 (q, ¹*J*(F,B) 40 Hz, 3F, B*F*₃). ¹¹B NMR (CD₃CN): δ = -0.68 (qt, ¹*J*(B,F) 41 Hz, ²*J*(B,F¹) 19 Hz).

IR $\bar{\nu}$ (cm⁻¹): 1361 w, 1315 m, 1272 m, 1221 vs, 1194 s, 1151 s, 1133 vs, 1067 s, 1038 vs, 988 m, 975 m, 854 w, 823 w, 797 m, 736 s, 694 m, 656 w, 619 w, 599 w, 569 w, 534 w.

Xenon difluoride (290 mg, 1.71 mmol) was added to the stirred suspension of K [C₆F₁₃BF₃] (700 mg, 1.64 mmol) in aHF (10 ml) at -30 °C. The reaction mixture was stirred for 2 h at -30 °C and for 1.5 h at 20 °C. The resulting solution contained unreacted K [C₆F₁₃BF₃] (¹⁹F NMR) (the resonance of XeF₂ interfered with the broad signal of aHF at -192 ppm).

Reaction of hexafluorobenzene with XeF_2 in aHF of different acidity

Xenon difluoride (48 mg, 0.28 mmol) was added in portions to the stirred mixture of C_6F_6 (53 mg, 0.28 mmol) in aHF (0.2 ml) at -15 °C. After each addition the reaction mixture was kept for several minutes at 20 °C until the evolution of xenon was completed. Octafluoro-1,4-cyclohexadiene was obtained in quantitative yield (¹⁹F NMR).

Xenon difluoride (53 mg, 0.31 mmol) was added in one portion to the stirred mixture of C_6F_6 (53 mg, 0.28 mmol) and K [HF₂] (71 mg, 0.91 mmol) in aHF (0.2 ml) at -15 °C. The mixture was kept at 20 °C for 30 minutes but there was no sign of a reaction. After 13 h only traces of octafluoro-1,4-cyclohexadiene were detected, besides unreacted C_6F_6 (¹⁹F NMR).

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