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(Fluoroorgano)fluoroboranes and -fluoroborates
Part 3. Polyfluoroalken-1-yldifluoroboranes

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Dedicated to Professor Voronkov on the occasion of his 80th birthday

Abstract

Polyfluoroalken-1-yldifluoroboranes $RCF{=}CFBF_2$ (R = F, *cis*-, *trans*-Cl, *trans*-C_4F_9, *cis*-C_2F_5, *cis*-C_6F_{13}, *trans*-C_4H_9, *trans*-C_6H_5) were prepared by defluoridation of the corresponding fluoroborate salts $K[RCF{=}CFBF_3]$ using boron trifluoride in dichloromethane or fluorotrichloromethane. The alkenylboranes were characterised by ^{1}H-, ^{11}B- and ^{19}F-NMR spectroscopy. Their reactivity towards ether, water and anhydrous HF was investigated.

Keywords: Boranes; Borates; Polyfluoroalken-1-yl boron compounds; NMR

1. Introduction

Recently we have described the successful defluoridation of the potassium salts of polyfluoroaryltrifluoroborates $K[C_6H_{5-n}F_nBF_3]$ ($n = 1–5$) and of perfluoroalkyltrifluoroborates $K[C_nF_{2n+1}BF_3]$ ($n = 3$, 6) under formation of the corresponding polyfluoroaryl- and perfluoroalkyldifluoroboranes using the fluoride anion acceptors BF_3 or arsenic pentafluoride [1,2]. In our previous paper [3], the synthesis of salts $K[RCF{=}CFBF_3]$ and their NMR and IR spectra are reported. Fluoro-containing alken-1-yldifluoroboranes are scarcely studied compounds. In 1960, trifluorovinyldifluoroborane was prepared from $CF_2{=}CFBCl_2$ and SbF_3 (59% yield) or from $(CF_2{=}CF)_2SnMe_2$ and BF_3 (18% yield) [4]. Later, the difluoroboranes $CF_2{=}C(CF_3)BF_2$ and *cis*-$BF_2CF{=}CFBF_2$ were obtained by the addition of BF_3 to $CF_2{=}C{=}CF_2$ (70% yield) [5] or B_3F_5 to $CF_2{=}CF_2$ (yield was not given) [6]. In a preliminary communication [7], we demonstrated the principal approach to polyfluoroalken-1-yldifluoroboranes based on the defluoridation of the corresponding $K[RCF{=}CFBF_3]$ salts using BF_3.

Here, we report the detailed synthesis of various difluoroboranes $RCF{=}CFBF_2$ (R = F, *cis*-, *trans*-Cl, *cis*-C_2F_5, *cis*-C_6F_{13}, *trans*-C_4F_9, *trans*-C_4H_9, *trans*-C_6H_5) by defluoridation of the fluoro borate salts in an inert solvent (CH_2Cl_2 or CCl_3F), their NMR spectra as well as their reactivities towards water, ether or anhydrous HF (aHF).

2. Results and discussion

Potassium polyfluoroalken-1-yltrifluoroborates, like the salts $K[C_6H_{5-n}F_nBF_3]$ and $K[C_nF_{2n+1}BF_3]$, are practically insoluble in weakly polar, weakly coordinating solvents [3]. BF_3 gas dissolves at low temperature in such solvents like CH_2Cl_2 or CCl_3F. Bubbling of BF_3 into the stirred suspension of potassium polyfluoroalken-1-yltrifluoroborate salts in CH_2Cl_2 or in CCl_3F at −40 to −60 °C led to the easy defluoridation under formation of the corresponding polyfluoroalken-1-yldifluoroboranes in high yields.

$$K[RCF=CFBF_3]_{(s)} + BF_{3(g)} \xrightarrow[-40\ to -\ 60°C]{CH_2Cl_2\ or\ CCl_3F} K[BF_4]_{(s)} + RCF=CFBF_2$$

R = F (**1**); *cis*-, *trans*-Cl (**2**); *cis*-C_2F_5 (**3**); *cis*-C_6F_{13} (**4**); *trans*-C_4F_9 (**5**); *trans*-C_4H_9 (**6**); *trans*-C_6H_5 (**7**).

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Table 1
The ^{11}B-NMR spectra of polyfluoroalken-1-yldifluoroboranes RCF=CFBF$_2$[a]

R	δ(B) (ppm)	$\tau_{1/2}$ (Hz)
F [8]	21.8 [b]	n.d.
cis-Cl	21.99	186
cis-C_2F_5	20.28	106
cis-C_6F_{13}	20.66	140
trans-Cl	21.10	175
trans-C_4F_9	20.04	156
trans-C_4H_9	21.72	160
[c]	22.6	n.d.

[a] Solutions in CH_2Cl_2. Substituents R are in *cis* or *trans* positions relative to the BF$_2$ group.

[b] In toluene-d_8.

[c] CH_2=$CHBF_2$ [9].

Defluoridation under the above conditions is neither accompanied by the migration of the C=C bond into the carbon chain nor by *cis*, *trans*-isomerisation of the products.

Alkenyldifluoroboranes **1**–**7** are well soluble either in CH_2Cl_2 or in CCl_3F at room temperature. Compounds **4** and **5** with the perfluorinated long alkyl chain separate at lower temperature forming compact oil phases. The solutions of boranes **1**–**5** are stable at 20 °C over days under a dry argon atmosphere, whereas the solutions of **6** and **7** are less stable and decompose at 20 °C within some hours to give BF$_3$, *trans*-RCF=CFH and additionally unrecognised fluoroorganics.

The ^{11}B-NMR spectra of the difluoroboranes **1**–**5** show the resonance of the boron atom at 21–22 ppm. This coincides with the previously reported ^{11}B-NMR data of CH_2=$CHBF_2$ and CH_2=$CFBF_2$ (Table 1) and is similar to those of fluoroaryldifluoroboranes [1] and perfluoroalkyldifluoroboranes [2]. In all cases, the ^{11}B-NMR signals are broad and unresolved ($\tau_{1/2} \approx 100$–190 Hz) due to the electric quadrupole of the three-coordinated boron atom (Table 1). The ^{19}F resonances of the fluorine atoms bonded to boron are located at -80 to -88 ppm and appear broad and unresolved regardless of the temperature (-40 to $+24$ °C) (Table 2). It should be noted that replacement of the fluorine atom in the *trans*-position to the BF$_2$ group by the substituents R = Cl, C_4H_9 and C_6H_5 has no effect on the δ(F) values of the fluorine atoms bonded at boron. The ^{19}F resonance of the BF$_2$ group in vinyldifluoroborane CH_2=$CHBF_2$ is also located in the same region at -89 ppm [11]. However, a remarkable deshielding of the fluorine atoms of the BF$_2$-group is observed in the ^{19}F-NMR spectra of the difluoroboranes *cis*-RCF=CFBF$_2$ (R = Cl, C_2F_5, C_6F_{13}) and *trans*-

Table 2
The ^{19}F-NMR spectra of polyfluoroalken-1-yldifluoroboranes RCF = CFBF$_2$[a]

R	δ(F) (ppm)				J (Hz)		
	F-1	F-2*cis*	F-2*trans*	BF$_2$	1,2*cis*	2*cis*,B*F*	2*cis*,2*trans*
F(-40 °C)	−207.19	−98.96	−72.06	−87.06	114	24	16
F [10] [b]	−206.6	−99.8	−72.8	−86.7	114	25	18
F (24 °C) [c]	Cl	−68.04	−55.98	n.d.	–	n.d.	n.d.
F (-40 °C) [c]	Cl	−68.09	−55.79	n.d.	–	n.d.	n.d.
trans-Cl (24 °C)	−171.64	−97.68	–	−86.12	134	24	–
trans-Cl (-40 °C)	−172.26	−97.92	–	−86.50	133	27	–
F [5] [d]	CF$_3$ [e]	−57.3	−47.6	−82.2	–	n.d.	39.0
trans-C_4H_9 (24 °C)	−184.86	−124.70	– [f]	−87.71	129	27	–
trans-C_4H_9 (-20 °C)	−184.88	−124.85	– [g]	−87.63	130	n.d.	–
trans-C_6H_5 (24 °C)	−178.88	−136.30	–	−87.00	128	n.d.	–
trans-C_4F_9 (24 °C)	−166.57	−149.08	– [h]	−82.97	123	n.d.	–
cis-Cl (24 °C)	−159.59	–	−68.77	−83.12	–	–	–
cis-C_6F_{13} (24 °C)	−146.27	– [i]	−123.31	−78.98	–	–	–
cis-C_2F_5 (24 °C)	−146.97	– [j]	−124.25	−80.36	–	–	–
cis-BF$_2$ [6]	−158	–	−158	−80.6	–	–	–

[a] Solutions in CH_2Cl_2. Fluorine atoms are in *cis* or *trans*-positions relative to the BF$_2$ group.

[b] In CFCl$_3$.

[c] Derived from an admixture (9%) of K[CF$_2$=CClBF$_3$].

[d] Neat liquid.

[e] δ(F): -59.9 (CF$_3$); J, Hz: (2*cis*,C*F*$_3$) 22.6, J, Hz: (2*trans*,C*F*$_3$) 12.0.

[f] J, Hz: (2,H-3) 25.

[g] δ(H): 2.61 (2 H-3), ~1.4 (2 CH$_2$), 0.86 (3 H-6).

[h] δ(F): -81.71 (3 F-6), -119.14 (2 F-3), -125.05, -126.92 (2 CF$_2$).

[i] δ(F): -81.48 (3 F-8), -116.79 (2 F-3), -122.49, -123.31 (3 CF$_2$), -126.68 (2 F-7).

[j] δ(F): -84.97 (3 F-4), -120.33 (2 F-3).

$C_4F_9CF{=}CFBF_2$. Similar phenomena were observed for *cis*-$BF_2CF{=}CFBF_2$ [6] and $CF_2{=}C(CF_3)BF_2$ [5]. Also the introduction of fluorine atoms in the *ortho*-positions of aryldifluoroboranes $C_6H_{5-n}F_nBF_2$ caused a similar effect [1]. It is likely, that this effect arises from an intramolecular fluorine–boron interaction.

Little information about the reactivity of fluoro-containing alkenyldifluoroboranes was known. It was reported that the interaction of borane **1** with water at room temperature resulted neither in carbon–boron nor in fluorine–boron bond cleavage but the products were not characterised. Alternatively, the quantitative protodeborylation of difluoroborane **1** took place at 120 °C (15 h) yielding trifluoroethylene [4]. The hydrolysis of compound $CF_2{=}C(CF_3)BF_2$ resulted in the polyfluorinated olefin $CF_2{=}CHCF_3$ (conditions were not reported) [5].

We have investigated the reactivity of some difluoroboranes towards water and ether and we have found that the treatment of difluoroboranes **1** and **3** with ether in CH_2Cl_2 gave as expected the etherates **8** and **9** in quantitative yield.

$$\underset{\mathbf{1}\ \text{or}\ \mathbf{3}}{RCF = CFBF_2} + OEt_2 \xrightarrow[-40°C\ \text{to r.t.}]{CH_2Cl_2} RC\mathit{F} = CFBF_2{\cdot}OEt_2$$

R = F (**8**); *cis*-C_2F_5 (**9**).

The coordination of diethyl ether (neutral *n*-electron donor) at the boron atom leads to a dramatic redistribution of the charge in the molecule which is reflected in the ^{19}F-NMR spectra. The spectrum of etherate **8** consists of four resonances at −94.32 (F-2*trans*), −118.82 (F-2*cis*), −199.71 (F-1) and −149.04 ($BF_2{\cdot}OEt_2$) ppm [*J*, Hz: (1,2*cis*) 114, (1,2*trans*) 17, (2*cis*,2*trans*) 73, (2*cis*, $BF_2{\cdot}OEt_2$) 14 and (F,B) 46]. The ^{19}F-NMR spectrum of compound **9** displays signals at −84.85 (3 F-4), −119.77 (2 F-3), −147.79 ($BF_2{\cdot}OEt_2$), −137.78 and −146.49 (F-1 and F-2*trans*) ppm. A comparison of the spectra of the adducts **8** and **9** with those of the starting difluoroboranes **1** and **3** (Table 2) and those of the corresponding salts K[$RCF{=}CFBF_3$] [3], demonstrates the dramatic changes when the BF_2 group [1] with the strongest σ- and π-electron accepting effect is converted to the neutral $BF_2{\cdot}OEt_2$ group with a tetra coordinated boron atom or to the negatively charged BF_3^- group with the strongest σ-electron donating and negligible mesomeric effect [1].

When CH_2Cl_2 solutions of $RCF{=}CFBF_2$ were reacted with water at 0 °C (R = F) or at ≥ −78 °C (R = *cis,trans*-Cl), the formation of the anions $[RCF{=}CFBF_3]^-$ [3] and $[BF_4]^-$ was detected in the aqueous phase (^{19}F-NMR). No ^{19}F-NMR signals which could be attributed to the alkenes $RCF{=}CFH$ or the boronic acids $RCF{=}CFB(OH)_2$ were observed neither in the aqueous nor in the organic phase. It is known from literature [4] that difluoroboranes $RCF{=}CFBF_2$ can dissolve BF_3. We have observed BF_3 after saturation of difluoroborane solutions at −40 °C (^{19}F), but BF_3 was not detected (^{19}F) after degasing. Admixtures of BF_3, which are still present, may be the additional source of HF during hydrolysis and responsible for the conversion of compounds **1** and **2** into $[RCF{=}CFBF_3]^-$ and the basis of $[BF_4]^-$ in the concurrent absence of $RCF{=}CFH$.

However, in anhydrous HF (aHF) the difluoroboranes **1** and **2** underwent protodeborylation and the corresponding fluoro-containing alkenes $RCF{=}CFH$ were formed. After shaking the $CF_2{=}CFBF_2$ solution in CH_2Cl_2 with aHF at −40 °C, the acidic phase contained both $CF_2{=}CFH$ and difluoroborane **1**. It is noteworthy to mention that the ^{19}F-NMR spectrum of the latter did not differ from that of $CF_2{=}CFBF_2$ in CH_2Cl_2. This fact verifies that no significant complex formation between trifluorovinyldifluoroborane and aHF takes place. A similar picture was observed during the reaction of difluoroborane **2** with aHF.

$$RCF = CFBF_2 + aHF \rightarrow RCF = CFH + BF_3$$

R = F; *cis*-, *trans*-Cl.

Alternatively, the addition of aHF to a solution of *trans*-$C_4F_9CF{=}CFBF_2$ in CH_2Cl_2 at −20 °C followed by warming up to room temperature did not result in the protodeborylation. The ^{19}F-NMR spectrum of the acidic phase showed resonances at −79.53 (3 F-6), −115.83 (2 F-3), −122.83, −124.50 (2 F-4 and 2 F-5), −159.83 (F-1) and −169.17 (F-2) ppm [*J*(1,2) 132 Hz], whereas the resonance of the fluorine atoms bonded to boron was not observed. The organic phase contained only traces of HF and the above-mentioned signals. This spectrum coincides with the spectrum obtained from K[*trans*-$C_4F_9CF{=}CFBF_3$] in aHF and differs from that of the salt in MeCN [resonances at −80.41, −115.95, −124.12, −125.60, −153.06 and −177.16 ppm, respectively, and at −144.03 (BF_3^-) ppm; *J*(1,2) 130 Hz] [3]. Presumably, in aHF the formation of a complex between difluoroborane **5** (Lewis acid) and HF (F-terminus acts as Lewis base) occurred. In fact, the observed ^{19}F-NMR spectrum can present an equilibrium between the neutral difluoroborane **5**, HF and the protonated perfluoro-*trans*-hexen-1-yltrifluoroborate.

$$trans\text{-}C_4F_9CF{=}CFBF_2 + 3HF \rightleftarrows [H_2F][trans\text{-}C_4F_9CF{=}CFBF_2(F{\cdots}HF)]$$

Taking into account the complex formation of **5** in aHF and the absence of a related complex of $CF_2{=}CFBF_2$ in aHF, the protodeborylation of difluoroboranes **1** and **2** can be described in terms of the direct attack of the proton on the carbon atom C-1 bonded to boron.

It should be noted that fluoroalkenes $RCF{=}CFH$ (R = F, *cis*-, *trans*-Cl) are significantly soluble in aHF at − 40 °C although their ^{19}F-NMR spectra show no protonation. For instance, the solution of CF_2=CFH in aHF (− 40 °C) displays resonances at − 99.69 (F-2*trans*), − 125.98 (F-2*cis*) and − 205.27 (F-1) ppm [*J*, Hz: (1,H) 70.9, (1,2*cis*) 116.7, (1,2*trans*) 33.0, (2*cis*,H) 3.4, (2*cis*,2*trans*) 88.4, (2*trans*,H) 12.6] (the positions of the fluorine atoms are with respect to the hydrogen atom). A solution of CF_2=CFH in CH_2Cl_2 (− 40 °C) is characterised by ^{19}F resonances at − 101.22 (F-2*trans*), − 127.04 (F-2*cis*) and − 206.27 (F-1) ppm [*J*, Hz: (1,H) 70.5, (1,2*cis*) 117.6, (1,2*trans*) 32.4, (2*cis*,H) 3.8, (2*cis*,2*trans*) 83.6, (2*trans*,H) 13.2] These spectral data coincide well with those taken from [12]. It is noteworthy, that the ^{19}F value δ(F-1) of CF_2CFH given in Ref. [13] is erroneous. This wrong value is cited in the books [14,15] and the new monograph [16].

3. Experimental

NMR spectra were measured on Bruker spectrometers WP 80 SY (^{1}H at 80.13 MHz and ^{19}F at 75.39 MHz) and Avance DRX 500 (^{1}H at 500.13 MHz, ^{11}B at 160.46 MHz and ^{19}F at 470.59 MHz). Shift values were reported with respect to TMS (^{1}H), $BF_3 \cdot OEt_2$ (^{11}B) and CCl_3F (^{19}F).

Dichloromethane, ether and CCl_3F were purified and dried by standard procedures. Hydrogen fluoride was dried by electrolysis (stainless steel cell, Ni electrodes). The preparation of salts K[RCF=$CFBF_3$] (R = F, *trans*-C_4F_9, *cis*-C_2F_5, *cis*-C_6F_{13}, *trans*-C_4H_9, *trans*-C_6H_5) and K[*cis*-, *trans*-ClCF=$CFBF_3$] (contains additionally 9% of K[CF_2=$CClBF_3$]) is described in Ref. [3].

All manipulations of moisture-sensitive compounds were performed in FEP (copolymer of tetrafluoroethylene and hexafluoropropylene) equipment under a dry argon atmosphere.

3.1. Preparation of RCF=$CFBF_2$ (general procedure)

Potassium polyfluoroalken-1-yltrifluoroborate K[RCF=$CFBF_3$] was suspended in CH_2Cl_2 or in CCl_3F at − 40 to − 65 °C and BF_3 (4–5 M excess) was bubbled for 20–60 min under stirring. The reaction mixture was flushed with dry argon to remove the excess of BF_3. After centrifugation at temperatures below − 40 °C (R = F, *cis*-, *trans*-Cl) or at − 20 °C (R = *trans*-C_4H_9) or at room temperature (r.t.) (R = *trans*-C_4F_9, *cis*-C_2F_5, *cis*-C_6F_{13}, *trans*-C_6H_5), the mother liquor was decanted, the precipitate was washed with CH_2Cl_2, and the extract was combined with the mother liquor. Yields of difluoroboranes RCF=$CFBF_2$ were higher than 80–90% (^{19}F-NMR with the internal quantitative references $CFCl_2CF_2Cl$ or $PhCF_3$).

3.2. Reaction of RCF=$CFBF_2$ with ether

A solution of RCF=$CFBF_2$ (R = F, *cis*-C_2F_5) (0.3–0.4 mmol) in CH_2Cl_2 (0.3–0.4 ml) was cooled to − 40 °C and anhydrous ether (1.7–2 M equivalents) was added. The solution was warmed to r.t. and showed the quantitative conversion of RCF=$CFBF_2$ to RCF=$CFBF_2 \cdot OEt_2$ (^{19}F-NMR).

3.3. Reactions of RCF=$CFBF_2$ with water

A. Ice water (0.4 ml) was added to a solution of CF_2=$CFBF_2$ (0.42 mmol) in CH_2Cl_2 (0.48 ml) at 0 °C and the mixture was shaken. The aqueous phase was decanted at 20 °C and diluted with D_2O (0.3 ml). The ^{19}F-NMR spectrum showed resonances at − 150.61 ppm ([H_3O] [BF_4]) and at − 98.53 (F-2*trans*), − 123.14 (F-2*cis*), − 199.00 (F-1) and − 142.70 (BF_3^-) ppm [*J*, Hz: (1,2*cis*) 110, (2*cis*,2*trans*) 87, (2*cis*,B*F*$_3$) 9, (F,B) 38] ([H_3O] [CF_2=$CFBF_3$]).

B. A solution of *cis*-, *trans*-ClCF=$CFBF_2$ (0.6 mmol) in CH_2Cl_2 (0.9 ml) was treated with aqueous acetone (0.3 ml; 0.08 mg of water) at − 78 °C. After warming to r.t. the organic phase contained no *cis*-, *trans*-ClCF=CFH (^{19}F-NMR). An additional amount of water (0.3 ml) was added and finally the water-containing phase was decanted. Its ^{19}F-NMR spectrum showed resonances of the anions $[BF_4]^-$ (− 151.07 ppm), [*cis*-ClCF=$CFBF_3]^-$ [− 102.02 (F-2*trans*), − 151.07 (F-1) and − 142.61 (BF_3^-) ppm], and [*trans*-ClCF=$CFBF_3]^-$ [− 126.80 (F-2*cis*), − 162.13 (F-1) and − 143.25 (BF_3^-) ppm; *J*, Hz: (1,B) 25, (1,2*cis*) 129, (2*cis*,B*F*$_3$) 9, (F,B) 40].

3.4. Reactions of RCF=$CFBF_2$ with aHF

A solution of RCF=$CFBF_2$ (R = F, *cis*-, *trans*-Cl, *trans*-C_4F_9) (0.2–0.4 mmol) in CH_2Cl_2 (0.3–0.8 ml) was cooled (− 40 to − 50 °C). Cold (− 40 °C) aHF (0.2–0.4 ml) was added under an argon atmosphere. After repeated shaking, the acidic phase was decanted into the pre-cooled (− 40 °C) FEP inliner and analysed by ^{19}F-NMR spectroscopy.

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