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Oxidation of Primary Aliphatic and Aromatic Aldehydes with Difluoro(aryl)-λ^3-bromane

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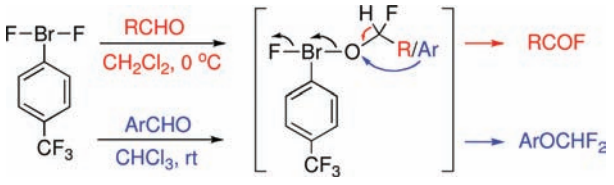
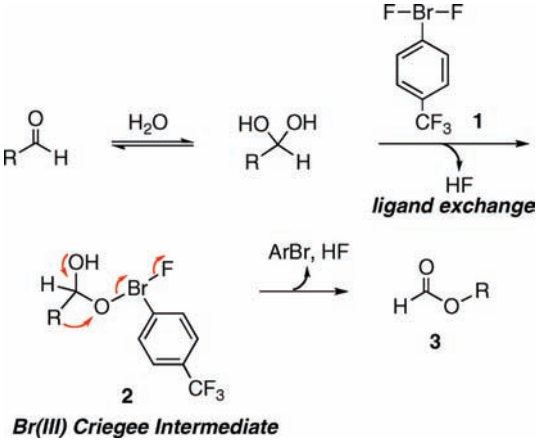
ABSTRACT



Oxidation of primary aliphatic aldehydes with *p*-trifluoromethylphenyl(difluoro)-λ^3-bromane in dichloromethane at 0 °C afforded acid fluorides selectively in good yields, while that of aromatic aldehydes in chloroform at room temperature produced aryl difluoromethyl ethers. A larger migratory aptitude of aryl groups compared to primary alkyl groups during a 1,2-shift from carbon to an electron-deficient oxygen atom in bromane(III) Criegee-type intermediates will result in these differences in the reaction courses.

The Baeyer–Villiger oxidation of ketones with peracids directly affords more complex and valuable esters or lactones.[1] Recently, we developed a conceptually distinct, modern strategy for the Baeyer–Villiger oxidation:[2] the method involves an initial hydration of water to carbonyl compounds, followed by ligand exchange of hypervalent Frohn reagent *p*-trifluoromethylphenyl(difluoro)-λ^3-bromane (**1**)[3] on bromane(III) with the resulting hydrate (*gem*-diol), yielding a new type of activated Criegee intermediate **2** as depicted in Scheme 1. The alkoxy-λ^3-bromane intermediate **2** undergoes Baeyer–Villiger rearrangement and produces rearranged ester **3** via facile reductive elimination of an aryl-λ^3-bromanyl group, because of the excellent hypernucleofugality.[4,5] This novel strategy makes it possible to selectively induce the Baeyer–Villiger oxidation of straight chain primary aliphatic as well as aromatic

Scheme 1



(1) (a) Baeyer, A.; Villiger, V. *Ber. Dtsch. Chem. Ges.* **1899**, *32*, 3625. (b) Hassall, C. H. *Org. React.* **1957**, *9*, 73. (c) Krow, G. R. *Org. React.* **1993**, *43*, 251. (d) Strukul, G. *Angew. Chem., Int. Ed.* **1998**, *37*, 1198. (e) Renz, M.; Meunier, B. *Eur. J. Org. Chem.* **1999**, 737. (f) Brink, G.-J.; Arends, I. W. C. E.; Sheldon, R. A. *Chem. Rev.* **2004**, *104*, 4105.

(2) Ochiai, M.; Yoshimura, A.; Miyamoto, K.; Hayashi, S.; Nakanishi, W. *J. Am. Chem. Soc.* **2010**, *132*, 9236.

(3) (a) Frohn, H. J.; Giesen, M. *J. Fluorine Chem.* **1998**, *89*, 59. (b) Ochiai, M.; Nishi, Y.; Goto, S.; Shiro, M.; Frohn, H. J. *J. Am. Chem. Soc.* **2003**, *125*, 15304. (c) Frohn, H.-J.; Hirschberg, M. E.; Wenda, A.; Bardin, V. V. *J. Fluorine Chem.* **2008**, *129*, 459.

(4) (a) Okuyama, T.; Takino, T.; Sueda, T.; Ochiai, M. *J. Am. Chem. Soc.* **1995**, *117*, 3360. (b) Ochiai, M. In *Chemistry of Hypervalent Compounds*; Akiba, K.-y., Ed.; Wiley-VCH: New York, 1999; Chapter 12. (c) Ochiai, M. In *Topics in Current Chemistry*; Wirth, T., Ed.; Springer: Berlin, 2003; Vol. 224, p 5.

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aldehydes, which is missing in the classical Baeyer–Villiger methods.[2]

The presence of a small amount of water plays a pivotal role in the λ^3-bromane-induced Baeyer–Villiger oxidation of carbonyl compounds: thus, rearranged formate **3** (R = *n*-C_9H_{19}) was produced in a good yield from the reaction of decanal in the presence of 1 to 10 equiv of water, while no formation of **3** was detected under anhydrous conditions.[2] We report herein an oxidation reaction of aldehydes with Frohn reagent **1** without adding external water under mild conditions (at 0 °C or ambient temperature): in the reaction of primary aliphatic aldehydes, carboxylic acid fluorides **4** were predominantly produced in good yields. In marked contrast, reaction of difluoro-λ^3- bromane **1** with aromatic aldehydes under anhydrous conditions afforded selectively rearranged difluoromethyl aryl ethers **7** in good yields.

Environmentally friendly hypervalent aryl-λ^3-iodanes (ArILL′) with two heteroatom ligands (L/L′) enjoy their rich chemistry, especially for oxidative transformations of various kinds of functionalities in modern organic synthesis.[6] On the other hand, oxidation reactions using hypervalent organo λ^3-bromanes (ArBrLL′) remain virtually unexplored,[7,8] although their oxidizing power seems to be greater than that of aryl-λ^3-iodanes.[9] In fact, the ionization potential of bromobenzene (8.98 eV) is larger compared to that of iodobenzene (8.69 eV).[10]

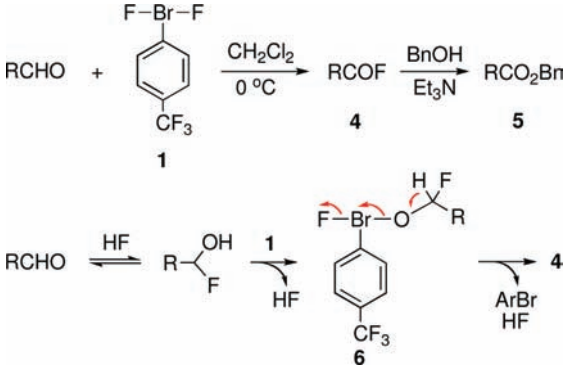
As noted above, in the presence of 2 equiv of water, reaction of decanal with difluoro-λ^3-bromane **1** (1.5 equiv) in dichloromethane at 0 °C for 1 h under argon resulted in the selective formation of Baeyer–Villiger product **3** (R = *n*-C_9H_{19}) in 80% yield, along with the formation of a small amount of decanoyl fluoride (**4a**) (4%) (Table 1, entry 3). In marked contrast, in the absence of water no rearranged formate ester **3** was produced by the reaction of decanal with **1**, but instead the reaction afforded moisture sensitive carboxylic acid fluoride **4a**[11] (73%, ^{1}H NMR yield) selectively (Scheme 2 and Table 1, entry 2). Acid fluoride **4a** showed a characteristic ^{19}F NMR signal at δ 44.9 ppm as a singlet in $CDCl_3$, which is in good agreement with the reported values for dodecanoyl (δ 44.92 ppm) and octanoyl fluoride (δ 44.9 ppm).[12] Further conversion to the known benzyl ester **5a** through treatment of the reaction mixture with benzyl alcohol/Et_3N[13] strongly suggests the formation of acid fluoride **4a** in the reaction.

Table 1. Oxidative Fluorination of Aliphatic Aldehydes with Difluoro-λ^3-bromane **1**[a]

				product		
entry	aldehyde	bromane **1** (equiv)	*t* (h)	**4**	yield (%)[b]	δ[c]
1	*n*-$C_9H_{19}CHO$	1.0	3	**4a**	37	
2	*n*-$C_9H_{19}CHO$	1.5	3	**4a**	73 (73)	44.9
3[d]	*n*-$C_9H_{19}CHO$	1.5	1	**4a**	4	
4[e]	*n*-$C_9H_{19}CHO$	1.5	3	**4a**	0	
5	MeCHO	1.5	3	**4b**	60 (50)	51.0
6	BuCHO	1.5	3	**4c**	68 (67)	44.8
7	*i*-BuCHO	1.5	3	**4d**	56 (42)	47.4
8	*n*-$C_5H_{11}CHO$	1.5	3	**4e**	69 (53)	45.0
9	$TsO(CH_2)_5CHO$	1.5	3	**4f**	81 (70)	45.0
10	$Cl(CH_2)_5CHO$	1.5	24	**4g**	81 (72)	44.9
11	$Br(CH_2)_5CHO$	1.5	36	**4h**	76 (63)	45.0
12	*t*-$BuCH_2CHO$	1.5	3	**4i**	25 (16)	53.9
13	*c*-$C_6H_{11}CHO$	1.5	4	**4j**	36 (36)	36.2
14	$PhCH_2CHO$	1.5	3	**4k**	trace[f]	44.4

[a] Conditions: aldehyde (0.05 M)/bromane **1**/CH_2Cl_2/0 °C/Ar. [b] ^{1}H NMR yields. Parentheses are isolated yields of benzyl esters **5**. [c] ^{19}F NMR chemical shifts (ppm, $CDCl_3$) of **4**. [d] Water (2 equiv) was used. Baeyer–Villiger product **3** (R = *n*-C_9H_{19}) was produced as a major product (80% yield). [e] Instead of difluoro-λ^3-bromane **1**, *p*-$CF_3C_6H_4IF_2$ was used. [f] $PhCH_2F$ (57%) and $PhCH_2OCHF_2$ (5%) were obtained.

Scheme 2



Difluoro-λ^3-bromane-induced oxidative fluorination of aliphatic aldehydes such as acetaldehyde, pentanal,

(5) Ochiai, M.; Tada, N.; Okada, T.; Sota, A.; Miyamoto, K. *J. Am. Chem. Soc.* **2008**, *130*, 2118.

(6) For reviews of aryl-λ^3-iodanes, see: (a) Merritt, E. A.; Olofsson, B. *Synthesis* **2011**, 517. (b) Uyanik, M.; Ishihara, K. *Chem. Commun.* **2009**, 2086. (c) Zhdankin, V. V.; Stang, P. J. *Chem. Rev.* **2008**, *108*, 5299. (d) Quideau, S.; Pouysegu, L.; Deffieux, D. *Synlett* **2008**, 467. (e) Ochiai, M.; Miyamoto, K. *Eur. J. Org. Chem.* **2008**, 4229. (f) Zhdankin, V. V. *Sci. Synth.* 2007, *31a*, Chapter 31.4.1, 161. (g) Wirth, T. *Angew. Chem., Int. Ed.* **2005**, *44*, 3656. (h) Tohma, H.; Kita, Y. *Adv. Synth. Catal.* **2004**, *346*, 111. (i) *Hypervalent Iodine Chemistry*; Wirth, T., Ed.; *Topics in Current Chemistry*; Springer: Berlin, 2003; Vol. 224. (j) Togo, H.; Sakuratani, K. *Synlett* **2002**, 1966. (k) Ochiai, M. In *Chemistry of Hypervalent Compounds*; Akiba, K., Ed.; VCH: New York, 1999; p 359. (l) Varvoglis, A. *The Organic Chemistry of Polycoordinated Iodine*; VCH: New York, 1992.

(7) (a) Nguyen, T. T.; Martin, J. C. *J. Am. Chem. Soc.* **1980**, *102*, 7382. (b) Nguyen, T. T.; Wilson, S. R.; Martin, J. C. *J. Am. Chem. Soc.* **1986**, *108*, 3803.

(8) For reactions of bromine trifluoride BrF_3, see: (a) Rozen, S. *Acc. Chem. Res.* **2005**, *38*, 803. (b) Hagooly, Y.; Rozen, S. *J. Org. Chem.* **2008**, *73*, 6780. (c) Cohen, O.; Sasson, R.; Rozen, S. *J. Fluorine Chem.* **2006**, *127*, 433. (d) Sasson, R.; Rozen, S. *Org. Lett.* **2005**, *11*, 2177.

(9) For reviews of aryl-λ^3-bromanes, see: (a) Ochiai, M.; Miyamoto, K.; Hayashi, S.; Nakanishi, W. *Chem. Commun.* **2010**, *46*, 511. (b) Ochiai, M. *Synlett* **2009**, 159. (c) Farooq, U.; Shah, A. A.; Wirth, T. *Angew. Chem., Int. Ed.* **2009**, *48*, 1018.

(10) *CRC Handbook of Chemistry and Physics*; Lide, D. R., Ed.; CRC: Boca Raton, FL, 1992.

(11) Lal, G. S.; Pez, G. P.; Pesaresi, R. J.; Prozonic, F. M. *Chem. Commun.* **1999**, 215.

(12) Oxidation of primary aliphatic alcohols with BrF_3 afforded a mixture of acyl fluorides (major products) and esters. See: Rozen, S.; Ben-David, I. *J. Fluorine Chem.* **1996**, *76*, 145.

(13) Chen, C.; Chien, C.-T.; Su, C.-H. *J. Fluorine Chem.* **2002**, *115*, 75.

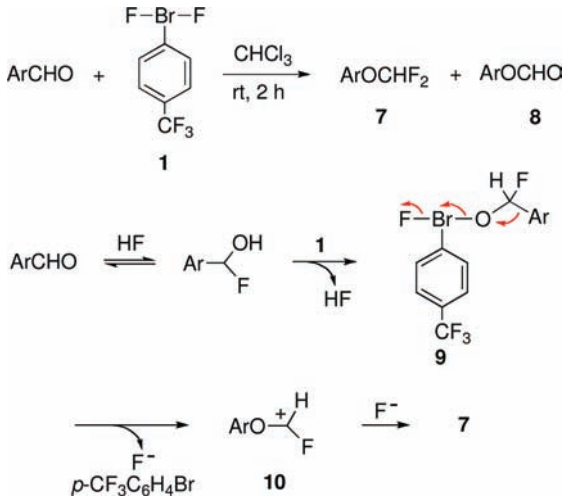
hexanal, and substituted hexanals with an electron-withdrawing tosyloxy (TsO), chloro, or bromo functionality at the terminal methyl group under anhydrous conditions afforded good yields (56−81%) of acyl fluorides **4b**−**h** selectively (entries 5−11). In marked contrast, *tert*-butylacetaldehyde, cyclohexanecarboxaldehyde, and phenylacetaldehyde resulted in the formation of very low to modest yields (3−36%) of fluorination products **4i**−**k** (entries 12−14). This low efficiency for the oxidative fluorination of these aldehydes, yielding acid fluoride **4**, will be due to the occurrence of a competing Baeyer−Villiger-type rearrangement, being evoked by the reported greater migratory aptitude of neopentyl, cyclohexyl, and benzyl groups compared to that of simple linear primary alkyl groups.[1d,14] In fact, formation of rearranged products was detected in the difluoro-λ^3-bromane-induced oxidative fluorination: for instance, in the reaction of phenylacetaldehyde both benzyl fluoride in a large amount (57%) and benzyl difluoromethy ether (5%) were produced (entry 14). The excellent hypernucleofugality of aryl-λ^3-bromanyl groups[5] suggests that the formation of these fluorine compounds probably involves a Baeyer−Villiger-type 1,2-shift of the benzyl group from a carbon to oxygen atom (Supporting Information, Scheme S1).[15]

Instead of Frohn reagent **1**, use of difluoro(aryl)-λ^3-iodane *p*-$CF_3C_6H_4IF_2$ did not undergo oxidation of decanal to acid fluoride **4a** under our conditions: thus, the aldehyde (98%) and a large amount of difluoro-λ^3-iodane were recovered unchanged, indicating the higher activity of the hypervalent difluoro-λ^3-bromane **1** in the oxidation of decanal (entry 4). Acid fluorides are generally prepared by a halogen exchange reaction of acyl chlorides or by fluorination of carboxylic acids.[16] We showed that λ^3-bromane-induced oxidative fluorination of simple linear primary aldehydes provides a new direct method for access to acid fluorides.[17]

A reaction pathway involving the initial formation of α-fluorohydrin from aldehyde through the addition of HF generated, in situ, its facile ligand exchange with difluoro-λ^3-bromane **1** on bromane(III), and finally reductive β-elimination of the resulting α-fluoroalkoxy(fluoro)-λ^3-bromane **6** will reasonably explain the selective formation of acid fluoride **4** from aldehyde (Scheme 2). In a close parallel to this, a reaction sequence consisting of the ligand exchange of an alcohol with Frohn reagent **1**, followed by the reductive β-elimination of intermediate alkoxy-λ^3-bromane, has been proposed for the oxidation of ethanol to acetaldehyde with **1**.[18]

In marked contrast to the reaction of α-hydroxyalkoxy-λ^3-bromane **2** (R = *n*-C_9H_{19}), which predominantly undergoes a 1,2-shift of the alkyl group to afford rearranged Baeyer−Villiger ester **3** (Scheme 1 and Table 1, entry 3),[2] the reductive β-elimination pathway leading to the formation of acid fluoride **4a** constitutes a major reaction course for α-fluoroalkoxy-λ^3-bromane **6** (R = *n*-C_9H_{19}, entry 2). The presence of the electron-withdrawing α-fluorine atom with a Hammett substituent constant σ_p of 0.06 in alkoxy-λ^3-bromane **6**,[19] instead of the electron-donating α-hydroxy group (σ_p = −0.37) in **2**, probably not only slows down the rate of the 1,2-shift of the alkyl group, yielding formate **3**, but also enhances the rate of reductive β-elimination producing **4a**, because of the increased acidity of the α-hydrogen atom in **6**.

Scheme 3



λ^3-Bromane-induced Baeyer−Villiger oxidation of benzaldehydes in the presence of a small amount of water exclusively produced aryl formates **8** in good to excellent yields.[2] In marked contrast to the reaction of aliphatic aldehydes (Table 1), even in the absence of water the reactions of benzaldehydes with difluoro-λ^3-bromane **1** hold their tendency to undergo Baeyer−Villiger-type oxidative rearrangement, but the major products in the reactions were changed from aryl formates **8** to aryl difluoromethyl ethers **7** (Scheme 3). Thus, reaction of benzaldehyde with Frohn reagent **1** (1.3 equiv) in chloroform at room temperature afforded difluoromethyl phenyl ether (**7a**)[20] (85%) as a major product and a small amount of formate ester **8a** (11%). Difluoromethyl ether **7a** is rather labile toward hydrolysis, and the pure sample of **7a** was obtained by

(14) Winnik, M. A.; Stoute, V. *Can. J. Chem.* **1973**, *51*, 2788.

(15) For the 1,2-shift of aryl groups from a carbon to oxygen atom in benzyloxy-λ^3-bromanes, see: Ochiai, M.; Yoshimura, A.; Miyamoto, K. *Tetrahedron Lett.* **2009**, *50*, 4792.

(16) (a) Carpino, L. A.; Beyermann, M.; Wenschuh, H.; Bienert, M. *Acc. Chem. Res.* **1996**, *29*, 268. (b) Lal, G. S.; Pez, G. P.; Pesaresi, R. J.; Prozonic, F. M.; Cheng, H. *J. Org. Chem.* **1999**, *64*, 7048. (c) Olah, G. A.; Nojima, M.; Kerekes, I. *Synthesis* **1973**, 487. (d) White, J. M.; Tunoori, A. R.; Turunen, B. J.; Georg, G. I. *J. Org. Chem.* **2004**, *69*, 2573.

(17) Radical oxidation of aldehydes with cesium fluoroxysulfate ($CsSO_4F$) in acetonitrile has been reported to give acid fluorides. See: (a) Stavber, S.; Planinsek, Z.; Zupan, M. *J. Org. Chem.* **1992**, *57*, 5334. (b) Stavber, S.; Kosir, I.; Zupan, M. *J. Org. Chem.* **1997**, *62*, 4916.

(18) Ochiai, M.; Yoshimura, A.; Mori, T.; Nishi, Y.; Hirobe, M. *J. Am. Chem. Soc.* **2008**, *130*, 3742.

(19) Hansch, C.; Leo, A.; Taft, R. W. *Chem. Rev.* **1991**, *91*, 165.

(20) (a) Zhang, L.; Zheng, J.; Hu, J. *J. Org. Chem.* **2006**, *71*, 9845. (b) Langlois, B. R. *J. Fluorine Chem.* **1988**, *41*, 247.

(21) Reichardt, C. *Solvents and Solvent Effects in Organic Chemistry*; Wiley-VCH: Weinheim, 2003.

preparative GC. Benzaldehydes with electron-donating (*o*- and *p*-Me, and *p*-MeO) and moderately electron-withdrawing substituents (*p*-F, *p*-Cl, and *p*-Br) selectively afforded aryl difluoromethyl ethers **7b**−**g** in good to high yields (Table 2, entries 2−7). The greater migratory aptitude of these aryl groups compared to that of linear primary alkyl groups will be responsible for the selective 1,2-aryl shift from a carbon to oxygen atom in α-fluorobenzyloxy-λ^3-bromanes **9**, instead of their reductive β-elimination of the aryl-λ^3-bromanyl group to give aromatic acid fluorides.[1]

Table 2. Oxidative Rearrangement of Aromatic Aldehydes with Difluoro-λ^3-bromane **1**[a]

		product, yield (%)[b]				
entry	aldehyde	**7**		δ^c	**8**	
1	PhCHO	**7a**	85	−81.1	**8a**	11
2	*o*-MeC_6H_4CHO	**7b**	66	−80.5	**8b**	17
3	*p*-MeC_6H_4CHO	**7c**	84	−80.9	**8c**	6
4	*p*-$MeOC_6H_4CHO$	**7d**	66	−80.9	**8d**	4
5	*p*-FC_6H_4CHO	**7e**	88	−81.5	**8e**	12
6	*p*-ClC_6H_4CHO	**7f**	89	−81.7	**8f**	9
7	*p*-BrC_6H_4CHO	**7g**	84	−81.7	**8g**	–
8[d]	*p*-$NO_2C_6H_4CHO$	**7h**	26	−82.5	**8h**	–
9[d,e]	*p*-$NO_2C_6H_4CHO$	**7h**	27	–	**8h**	–
10[d,e,f]	*p*-$NO_2C_6H_4CHO$	**7h**	20	–	**8h**	–

[a] Conditions: aldehyde (0.05 M)/bromane **1** (1.3 equiv)/$CHCl_3$/room temperature/2 h/Ar. [b] ^1H NMR yields. [c] ^{19}F NMR chemical shifts (ppm, $CDCl_3$) of **7**. [d] Yields of acid fluoride *p*-$NO_2C_6H_4COF$: 41% (entry 8), 65% (entry 9), and 70% (entry 10). [e] In CH_2Cl_2 as a solvent. [f] Bromane **1** (1.5 equiv) and 2,6-di-*tert*-butylpyridine (0.1 equiv) were used.

In fact, *p*-nitrobenzaldehyde with a highly electron-deficient aryl group, exhibiting a poor migratory aptitude, produced a large amount of acid fluoride *p*-$NO_2C_6H_4COF$ (41%) at the expense of the formation of difluoromethyl ether **7h** (26%) (entry 8). Changing the solvent from chloroform to dichloromethane increased the yield of the acid fluoride to 65% (entry 9).

Chloroform with a larger solvent acceptor number (*AN*) of 23.1 than that of dichloromethane (20.4)[21] more efficiently solvates and deactivates an anionic species such as the fluoride anion, which probably participates in the benzylic hydrogen abstraction of α-fluoro-*p*-nitrobenzyloxy-λ^3-bromane **9h** during reductive β-elimination, leading to the formation of *p*-$NO_2C_6H_4COF$. Thus, solvent hydrogen bonding toward the fluoride anion will be more effective in chloroform solution than in dichloromethane solution, which was further supported by the fact that the former is more acidic with an estimated pK_a value of ca. 24.[22] Therefore, the rate of reductive β-elimination of **9h**, compared to that of the oxidative 1,2-aryl shift, will be slowed down more remarkably in chloroform solution.[15] For this reason, dichloromethane was used as a solvent in the λ^3-bromane-induced oxidative fluorination of primary aliphatic aldehydes to acyl fluorides **4** (Table 1), where the fluoride anion (or hydrogen fluoride) may participate in the reductive β-elimination of α-fluoroalkoxy-λ^3-bromane **6**. Addition of a small amount of 2,6-di-*tert*-butylpyridine slightly increased the rate of β-elimination of α-fluorobenzyloxy-λ^3-bromane **9h** and afforded the acid fluoride in 70% yield (entry 10).[23]

Stavber and co-workers reported oxidative rearrangement of benzaldehydes to aryl difluoromethyl ethers **7** using xenon difluoride in the presence of large excessive amounts (4−5 equiv) of hydrogen fluoride, where initial formation of α-fluorohydrins via the addition of hydrogen fluoride to aldehydes and the subsequent ligand exchange of xenon difluoride with formation of alkoxyxenon fluoride were proposed.[24]

Thus, the reaction of aldehydes with difluoro-λ^3-bromane **1** seems to involve the initial formation of α-fluorohydrins and then their ligand exchange reaction on Br(III) of **1**. The follow-up reaction is dependent on the magnitude of the migratory aptitude of substituents: thus, α-fluorobenzyloxy-λ^3-bromanes **9** mostly undergo a 1,2-shift of aryl groups with a high migratory aptitude to give aryl difluoromethyl ethers **7**, while in α-fluoroalkoxy-λ^3-bromanes **6** reductive β-elimination leading to the formation of acid fluorides **4** constitutes a major reaction pathway, because of the decreased migratory aptitude of alkyl groups.

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Supporting Information Available. Typical experimental procedures and spectral data. This material is available free of charge via the Internet at http://pubs.acs.org.

(22) (a) Margolin, Z.; Long, F. A. *J. Am. Chem. Soc.* **1973**, *95*, 2757. (b) Bohme, D. K.; Lee-Ruff, E.; Young, L. B. *J. Am. Chem. Soc.* **1972**, *94*, 5153.

(23) Addition of an equimolar amount of 2,6-di-*tert*-butylpyridine completely inhibited the oxidation of *p*-nitrobenzaldehyde. This is probably due to deactivation of difluoro-λ^3-bromane **1** through the coordination of the pyridine base to the hypervalent bromane(III) with formation of a tetracoordinated λ^3-bromane species.

(24) (a) Stavber, S.; Koren, Z.; Zupan, M. *Synlett* **1994**, 265. (b) Tius, M. A. *Tetrahedron* **1995**, *51*, 6605. (c) Tamura, M.; Takagi, T.; Quan, H.; Sekiya, A. *J. Fluorine Chem.* **1999**, *98*, 163.