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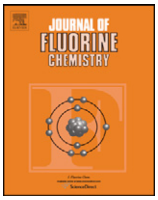

Review

1,2-Dibromotetrafluoroethane (Freon 114B2) as a building block for fluorine compounds

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ABSTRACT

This review provides an overview of several synthetic applications of 1,2-dibromotetrafluoroethane leading to convenient preparation of numerous fluorine compounds containing –CF_2CF_2– moiety *via* ionic and free radical reactions.

Contents

1. Introduction

In the preceding paper [1] numerous applications of 1-bromo-1-chlorotrifluoroethane (Halothane) as a building block for the synthesis of fluorine compounds have been summarized. Another synthetically useful, readily available and not expensive compound for such purpose is 1,2-dibromotetrafluoroethane. This compound is a commercial product, known as Freon 114B2 and Halon 2402, with a number of applications, *e.g.* fire extinguisher, refrigerant, leak detector, and others. It is colorless, inflammable, volatile liquid (b.p. 47.2 °C) easily made by the light promoted addition of bromine to tetrafluoroethylene [2].

1,2-Dibromotetrafluoroethane has a number of advantages to be useful building block for the synthesis of fluorine containing compounds. They are:

- active, removable bromine atoms
- conveniently low boiling point which allows easy removal of an excess of the reagent from the reaction mixture
- non-flammability
- low cost

The above factors caused an interest of 1,2-dibromotetrafluoroethane as a reagent in organofluorine chemistry and a number of applications have been reported till nowadays.

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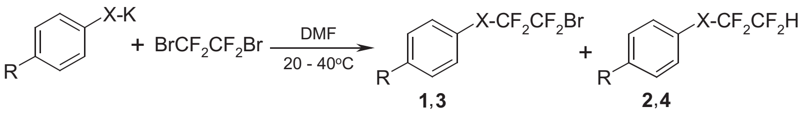
2. Reactions with phenoxides and thiophenoxides

The first announcement on the synthetic application of 1,2-dibromotetrafluoroethane was that by Rico and Wakselman [3] who reported its reactions with potassium thiophenoxides in dimethylformamide (DMF) leading to mixtures of aryl 2-bromotetrafluoroethyl thioethers (**1**) and their reduction products, 1,1,2,2-tetrafluoroethyl thioethers (**2**). Similarly, reactions with potassium phenoxides gave mixtures of 2-bromotetrafluoroethyl ethers (**3**) and 1,1,2,2-tetrafluoroethyl ethers (**4**). The total yields and the proportions of **1** to **2** and **3** to **4** sharply decreases with the decreasing nucleophilicity of the respective thiophenoxides and phenoxides.



1,**3** **2**,**4**

	X = S		X = O	
R	Yield (%)	**1** : **2**	Yield (%)	**3** : **4**
CH_3	50	50:50	40	95:5
H	35	40:60	28	93:7
Cl	30	75:25	24	83:17
NO_2	5	15:85	15	38:62

In the case of phenoxides addition of small amount of mercaptan was necessary to initiate the reaction and therefore an ionic chain mechanism was postulated for the above reactions as depicted below [3]. In this scheme $BrCF_2CF_2Br$ is both a brominating agent and tetrafluroethylene precursor.

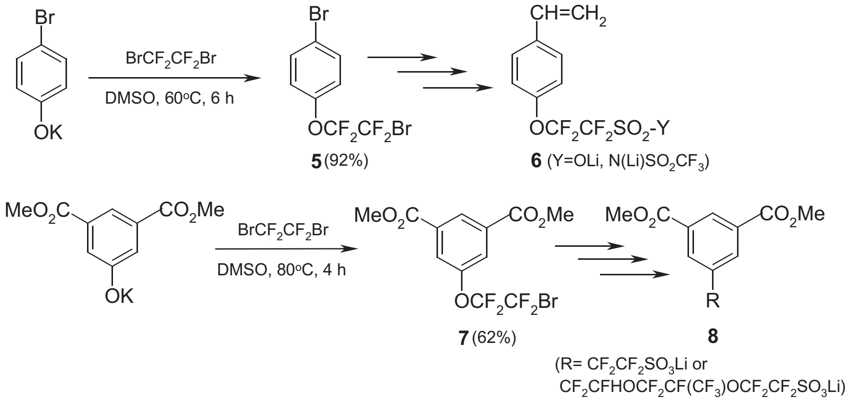
Initiation : $RS^- + BrCF_2CF_2Br \rightarrow RSBr + {}^-CF_2CF_2Br$
R = AR-for thiophenoxides and n-C_3H_7-
for phenoxides

Propagation : $CF_2CF_2Br^- \rightarrow CF_2{=}CF_2 + Br^-$
$\underset{(X=O,S)}{ArX^-} + CF_2{=}CF \rightarrow ArXCF_2CF_2^-$

$ArXCF_2CF_2^- + BrCF_2CF_2Br \rightarrow ArXCF_2CF_2Br + {}^-CF_2CF_2Br$

Termination : $ArXCF_2CF_2^- + \text{“H”}_{solvent} \rightarrow ArXCF_2CF_2H$

Much better results in favour of 2-bromotetrafluoroethyl ethers of the type (**3**), even in absence of mercaptans, were achieved by maintaining strictly anhydrous conditions. Thus the reaction of $BrCF_2CF_2Br$ with carefully dried potassium salt of 4-bromophenol in dry dimethylsulphoxide (DMSO) afforded the appropriate 2-bromotetrafluoroethyl ether **5** as the sole product in a 92% yield. Under the same conditions the reaction with potassium dimethyl 5-hydroxyisophtalate gave ether **7** of 93% purity and after TLC isolation of a 62% yield [4]. Compounds **5** and **7** were used as precursors for the synthesis of styrene and dimethylisophtalate monomers **6** and **8** with pendant lithium fluoroalkylsulphonate or sulphonimide functional groups [4].



5 (92%)

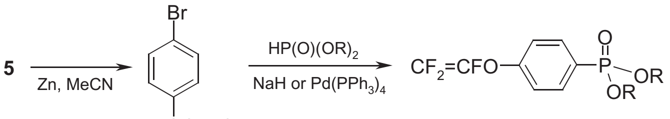
6 (Y=OLi, $N(Li)SO_2CF_3$)

7 (62%)

8

(R= $CF_2CF_2SO_3Li$ or
$CF_2CFHOCF_2CF(CF_3)OCF_2CF_2SO_3Li$)

2-Bromotetrafluoroethyl ether **5**, after reduction with zinc to the corresponding trifluorovinyl ether **9** followed by treatment with trimethyl or triethyl phosphite, was transformed into perfluorovinyl ether monomers **10** containing phosphonic acid functionality. These monomers were claimed to be potentially useful for proton exchange membranes for fuel cells [5].



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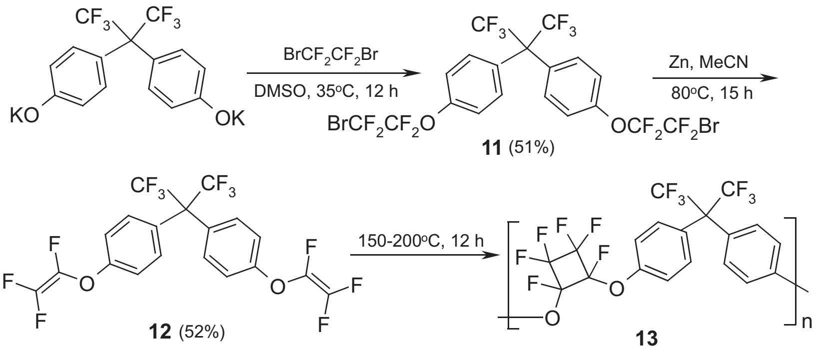
10 (R = Me, Et)

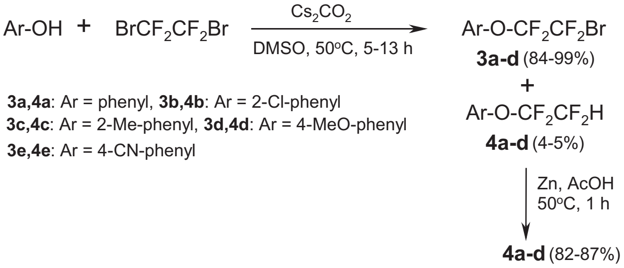
Fluoroalkylation of potassium salt of 2,2-bis(4-hydroxyphenyl)-1,1,1,3,3,3-hexa-fluoropropane with $BrCF_2CF_2Br$ gave ether **11** which, by the zinc mediated elimination afforded bis(trifluorovinyloxyphenyl) monomer **12**. Heating of the latter at

180–220 °C gave semi-crystalline polymer **13** with high molecular weight [6].

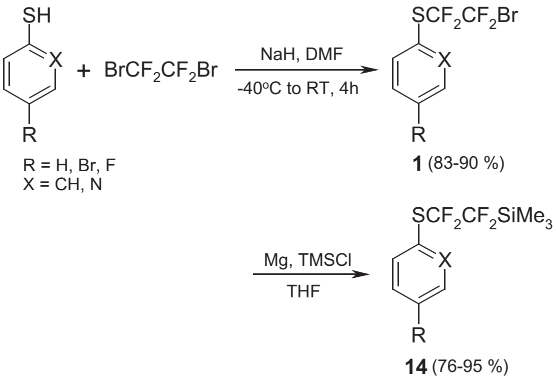


Synthesis of compounds of the type **11**, **12** and finally polymers of the structure analogical to **13** was earlier reported starting from carefully dried potassium salts of resorcinol, 3-trifluoromethylphenol, 4,4′-dihydroxybiphenyl, and 1,1,1-tris(4-hydroxy-phenyl)ethane [7].

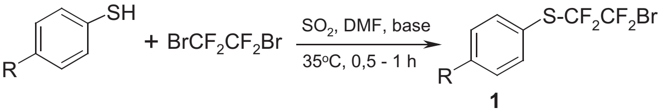
Preparation of various aryl tetrafluoroalkyl ethers by nucleophilic attack on 1,2-dibromotetrafluoroethane has been much improved by generating phenoxides *in situ*. Using anhydrous potassium, or preferably, cesium carbonate as a base in dry DMSO the substitutions were completed in short time to afford high yields of the desired 2-bromotetrafluoroethyl ethers **3a–d** with only trace amounts of the bromine reduction products **4a–d**. Reduction of compounds **3a–d** with zinc dust in acetic acid gave aryl tetrafluoroethyl ethers **4a–d** [8].



Similarly, when thiophenoxides were generated *in situ* using sodium hydride as a base, high yields of 2-bromotetrafluoroethyl thioeters of the type **1** were obtained. Treatment of these products with trimethylsilyl chloride (TMSCl) and magnesium afforded arylthio-(1,1,2,2-tetrafluoroethyl)trimethyl silanes (**14**) [9]. Compounds **14** served as tandem anions and radical tetrafluoroethylene synthons for the introduction of $–CF_2CF_2–$ moiety. Starting from carbonyl compounds, the preparation of tetrafluoroethyl substituted alcohols and tetrafluorotetrahydropyrans has been reported [10].



Fluoroalkylation of thiophenols with $BrCF_2CF_2Br$, but not thiophenolates, promoted by sulphur dioxide and aromatic amines in dimethylformamide (DMF) has been recently reported [11]. The reactions with thiophenol and thiocresol preceded selectively to give aryl 2-bromotetrafluoroethyl thioethers (**1**) in high yields (up to 99%) and as the only products without formation of the bromine reduction products of the type **2**. The best results were obtained in the presence of organic bases, γ-collidine, 2,5,-lutidine and β-picoline. Electron-withdrawing substituents in the aromatic ring caused sharp decrease in the yield of **1** (22% for NO_2).



Base	R = H Yield (%) of **1**	R = CH_3 Yield (%) of **1**
pyridine		78
beta-picoline	63.5	88
2,5-lutidine		98
gamma-collidine		99

The absence of hydrogenated thioethers in the products of these reactions, even in the presence of water and sharp lowering the yields of ethers **1** by the addition of free radical traps such as *p*-dinitrobenzene to the reaction mixture, led the Authors to the assumption then these reactions follow a radical pathway. Also, formation of small amounts of tetrafluoroethylene was detected in few cases. A $S_{RN}1$ type mechanism in which at the first step a single electron transfer takes place from the complexes of thiophenol with an amine was supposed [11].

$$XC_6H_4S^- + SO_2 \rightarrow XC_6H_4S^{\bullet} + SO_2^{\bullet-}$$

$$SO_2^{\bullet-} + BrCF_2CF_2Br \rightarrow SO_2 + [BrCF_2CF_2Br]^{\bullet-} \rightarrow {}^{\bullet}CF_2CF_2Br + Br^-$$

$$XC_6H_4S^- + {}^{\bullet}CF_2CF_2Br \rightarrow [XC_6H_4SCF_2CF_2Br]^{\bullet-}$$

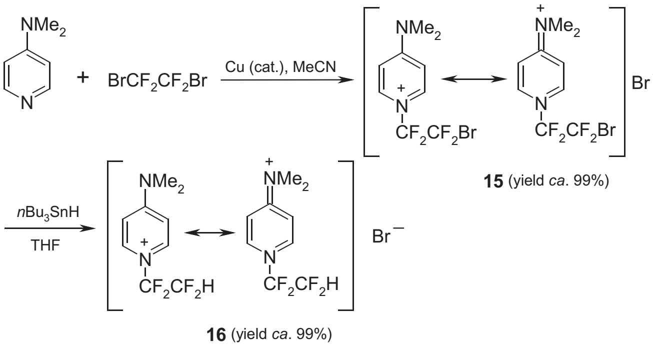
$$[XC_6H_4SCF_2CF_2Br]^{\bullet-} + SO_2 \rightarrow XC_6H_4SCF_2CF_2Br + SO_2^{\bullet-}$$

$$[XC_6H_4SCF_2CF_2Br]^{\bullet-} + BrCF_2CF_2Br \rightarrow SO_2 + Br^- + CF_2{=}CF_2$$

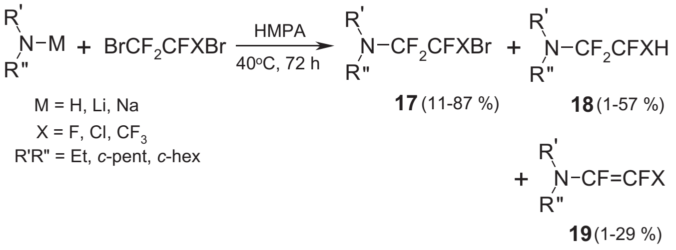
$$SO_2^{\bullet-} + {}^{\bullet}CF_2CF_2Br \rightarrow SO_2 + Br^- + CF_2{=}CF_2$$

3. Reactions with amines

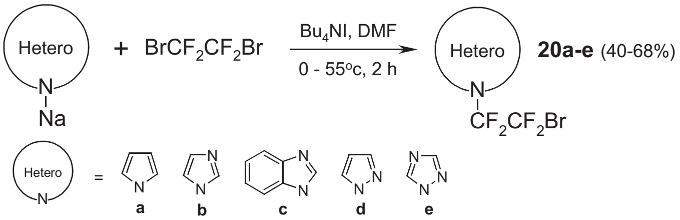
1,2-Dibromotetrafluoroethane and 4-dimethylaminopyridine reacted in polar, aprotic solvents, *e.g.* acetonitrile, to furnish 1-(4-dimethylaminopyridinium)-2-bromo-1,1,2,2-tetrafluoroethane bromide (**15**). In the presence of catalytic amount of activated copper or zinc the yield of **15** was almost quantitative [12] while on UV irradiation, without a catalyst, only 30% yield was achieved [13]. The reaction was totally blocked in presence of *p*-dinitrobenzene and, therefore, a radical mechanism pathway was postulated for this reaction. Hydrogenation of **15** with *n*-tributyltin hydride in THF gave 1-(4-dimethylaminopyridinium)-1,1,2,2-tetrafluoroethane bromide (**16**) [12]. The molecular structures for both **15** and **16** were determined [13].



Aliphatic secondary amines and amides reacted with perfluorohaloalkanes BrCF$_2$CFXBr (X = F, Cl, CF$_3$) in hexamethylphosphoramide (HMPA) to afford *N*-perfluorohaloalkylated tertiary amines **17**, **18** and **19** in fair to good yields. Evidence for an anionic chain process initiated by the bromophilic attack of nitrogen nucleophiles on the C–Br bond has been provided. Unfortunately, the resulting *N*-perfluorohaloalkylated derivatives were found to hydrolyse readily which hindered the isolation of the pure compounds [14].

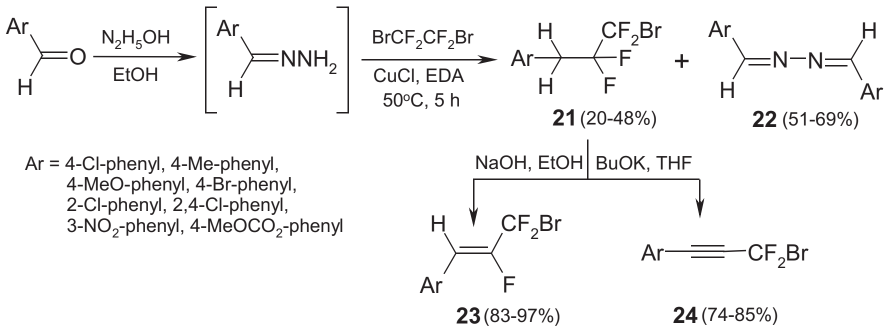


In contrary, nucleophilic substitution of BrCF$_2$CF$_2$Br with sodium salts of five-membered aromatic heterocycles afforded stable products, which were purified by distillation in vacuum or even at atmospheric pressure. Thus, reactions with pyrrole, imidazole, benzimidazole, pyrazole and triazole in DMF in the presence of catalytic amounts of tetrabutylammonium iodide afforded the respective *N*-(2-bromotetrafluoroethyl) derivatives **20a**–**e** in a 40–65% yields [15]. Compounds **20** reacted with thiophenols and aliphatic thiols in liquid ammonia under UV irradiation yielding the 2-heteroaryltetrafluoroethyl sulphides. The latter were oxidized to sulphones and by reactions with sodium dithionite converted to sodium salts of the corresponding sulphinic acids [15].



The bromine atom in compounds **20** was substituted with trimethylsilyl group by treatment with trimethylsilyl chloride providing an easy route to *N*-tetrafluoro(trimethylsilyl)ethyl azoles and by consecutive treatment of the latter with various electrophiles to the corresponding fluorinated carbinols, ketones, carboxylic acids and other derivatives [16].

Hydrazones of aromatic aldehydes reacted with BrCF$_2$CF$_2$Br under copper catalysis to give (3-bromo-2,2,3,3-tetrafluoropropyl)arenes (**21**) in moderate yields (20–48%) and *sym*-azines of carbonyl compounds (**22**) as side products. The reactions were conducted in ethanol and catalysed by copper chloride and 1,2-ethylenediamine [17]. Subsequent elimination of one molecule of HF from **21** by sodium hydroxide resulted in stereospecific formation of Z-isomers of styrene type alkenes (3-bromo-2,2,3-trifluoroprop-1-ene-1-yl)arenes (**23**). Treatment of **21** with more strong base such as potassium *tert*-butoxide lead to elimination of two molecules of HF and formation of alkynes bearing CF$_2$Br end group (3-bromo-3,3-difluoroprop-1-yn-1-yl)arenes (**24**). Both eliminations proceeded in high yields [17].

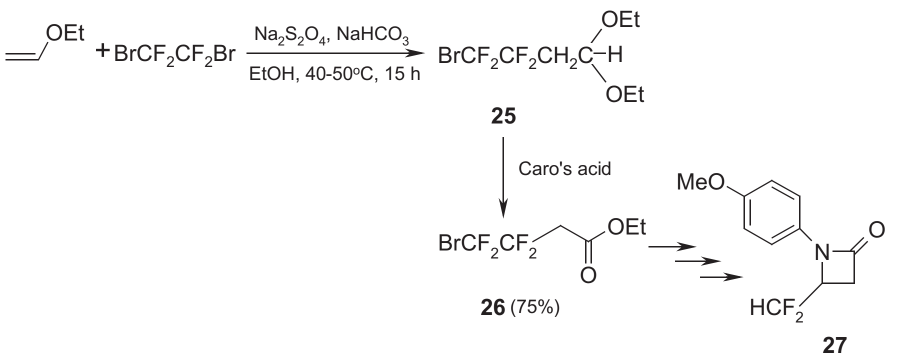


4. Reactions involving sulphinatodehalogenation of BrCF$_2$CF$_2$Br

Sulphinatodehalogenation of perfluoroalkyl halides, using sodium dithionite or related reagent as the free radical initiators in aqueous solutions, is the most convenient and general way of generating perfluoroalkyl radicals. In this reaction system, dithionite anions ($S_2O_4^{2-}$) exist in equilibrium with radical-anions $SO_2^{\bullet -}$, which by a SET process abstract halogens from perfluoroalkyl halides to generate perfluoroalkyl radicals $R_F^{\bullet}$. Reaction of these electron deficient radicals, generated from numerous per- and poly-fluorohaloalkanes with a variety of electron rich substrates have been widely described [18,19].

4.1. Reactions with alkenes and alkynes

1,2-Dibromotetrafluoroethane under sulphinatodehalogenation conditions (Na$_2$S$_2$O$_4$, NaHCO$_3$ and C$_2$H$_5$OH) generates the radical BrCF$_2$CF$_2^{\bullet}$ which reacts readily with alkenes, *e.g.* ethyl vinyl ether, affording high yields of bromofluoroalkyl diethyl acetates **25** [20]. Oxidation of ketones **25** with Caro's acid gave the respective esters **26** which by a number of transformation were converted to difluoromethylene β-lactams **27** [21].

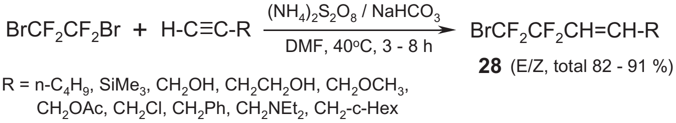


25

26 (75%)

27

Additions of $BrCF_2CF_2Br$ to various alkynes initiated by the ammonium peroxydisulphate–sodium formate system in DMF were reported. Under this conditions the addition products were instantaneously debrominated to give high yields of mixtures of the E and Z isomers of alkenes **28** terminated with the $BrCF_2CF_2$– group [22].



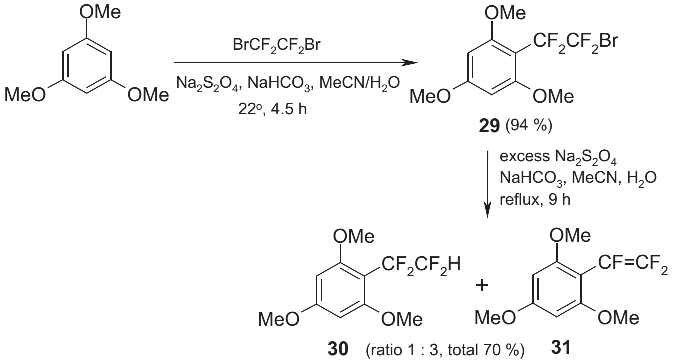
$$BrCF_2CF_2Br + H\text{-}C\equiv C\text{-}R \xrightarrow[\text{DMF, 40°C, 3 - 8 h}]{(NH_4)_2S_2O_8\ /\ NaHCO_3} BrCF_2CF_2CH{=}CH\text{-}R$$

28 (E/Z, total 82 - 91 %)

R = n-C_4H_9, $SiMe_3$, CH_2OH, CH_2CH_2OH, CH_2OCH_3, CH_2OAc, CH_2Cl, CH_2Ph, CH_2NEt_2, CH_2-c-Hex

4.2. Reactions with aromatics and heteroaromatics

The reaction of 1,2-dibromotetrafluoroethane with 1,3,5-trimethoxybenzene in the $Na_2S_2O_4/CH_3CN/H_2O$ system resulted in C-alkylation to give 1-(2-bromotetrafluoroethyl)-2,4,6-trimethoxynezene (**29**) almost quantitatively. The reaction proceeded readily at ambient temperature affording crude crystalline **29** which was pure enough to give correct elemental analysis without purification. Bromine atom in **29** was found to be fairly unreactive but further treatment with large excess of $Na_2S_2O_4$ in an acetonitrile–water system at reflux temperature resulted in reduction of the bromine and elimination of both Br and F radicals to give a mixture of 1-1,1,2,2-tetrafluoroethyl-2,4,6-trimethoxybenzene (**30**) and 1-(trifluoro-ethenyl)-2,4,6-trimethoxybenzene (**31**) in a 70% overall yield [23].

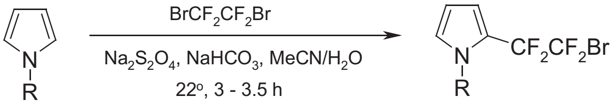


29 (94 %)

30 (ratio 1 : 3, total 70 %) **31**

The reaction with 1,2,3-trimethoxybenzene was much less successful; even when elevated temperature and prolonged reaction time were applied, only partial conversion occurred and a mixture containing much of unreacted trimetoxybenzene and all three isomeric alkylation products was formed [23]. Similarly, the reaction with mesitylene gave low yield of oily product containing mostly unreacted substrate, the expected (2-bromotetrafluoro-ethyl)mesitylene and crystalline dimeric product, derivative of hexamethyl-bicyclohexyl-tetraene (isolated yield *ca.* 13%) [23].

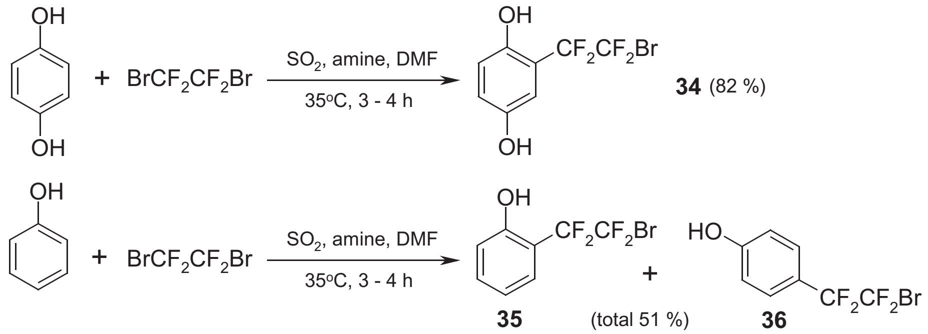
Pyrrole and *N*-methylpyrrole reacted with $BrCF_2CF_2Br$ under sulphinatodehalogenation conditions to give 2-(2-bromotetrafluoroethyl) substituted pyrroles **32** and **33** in good yields. Compound **32** was found to be rather unstable and quickly getting dark on contact with the air but *N*-methyl analog **33** was stable enough to be stored for a couple of weeks in the refrigerator [23].



32 R = H (73 %)

33 R = CH_3 (72 %)

An aromatic ring perfluoroalkylation of pyrrole and phenols occurred also when sulphur dioxide-organic base activating systems in DMF were applied. Using pyridine, β-picoline, 2,5-lutidine or γ-collidine as a base the reaction afforded fluoroalkylated pyrrole **32** in a 82% isolated yield. Under the same conditions, hydroquinone gave (2-bromotetra-fluoroethyl)hydroquinone (**34**) with 85% yield. The reaction with phenol was less efficient giving *ortho*- and *para*-substituted phenols **35** and **36** with total yield of 51% [24].

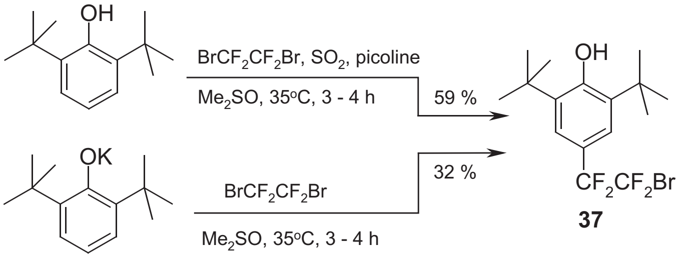


34 (82 %)

35 (total 51 %) **36**

Sulphur dioxide promoted reaction of $BrCF_2CF_2Br$ with sterically hindered 2,6-di-*tert*-butylphenol afforded 2,6-di-*tert*-butyl-4-(2-bromotetrafluoroethyl)phenol (**37**) in a 59% yield. It was shown that also the corresponding potasium phenolate undergo exclusively C-fluoroalkylation to give compound **37** as the only product albeit in much lower yield; no O-alkylation was observed.

A supposition was made that the monoelectronic reduction of 1,2-dibromotetrafluoroethane with sterically hindered phenolate anion can initiate the radical fluoroalkylation process [25].



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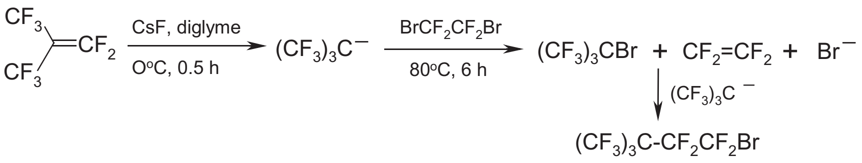
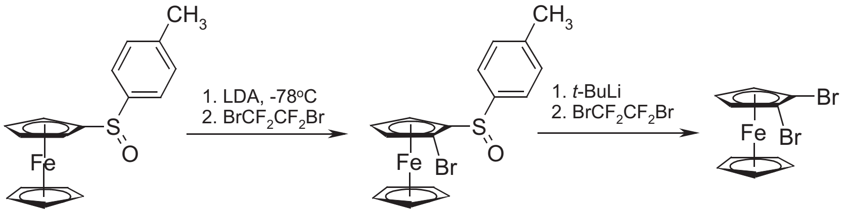
5. $BrCF_2CF_2Br$ as brominating agent

Perfluorocarbanion $(CF_3)_3C^-$, obtained by the reaction of perfluoroisobutene with caesium fluoride, easily substituted bromine in $BrCF_2CF_2Br$ to give perfluoroalkyl bromide **38**. The authors suggested that in the first step of this reaction undergoes bromination of the carbonanion to give *tert*-perfluorobutyl bromide, which then adds to simultaneously formed tetrafluoroethylene affording final product [26,27].



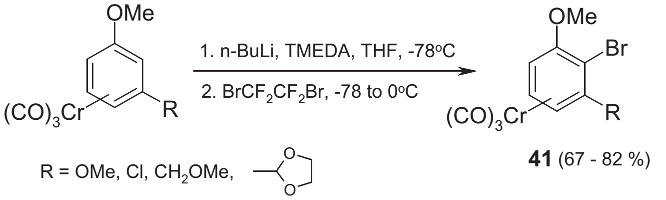
38 (52 %)

1,2-Dibromotetrafluoroethane was applied as electrophilic brominating agent for lithiated derivatives of aromatic compounds. *Ortho*-lithiation of *p*-tolylsulphinylferrocene followed by quenching with $BrCF_2CF_2Br$ gave 2-bromo(*p*-tolylsulphinyl)ferrocene (**39**) and subsequent lithiation of **39** followed by treatment with $BrCF_2CF_2Br$ afforded 1,2-dibromoferrocene **40** [28].



39 (84 %) **40** (86 %)

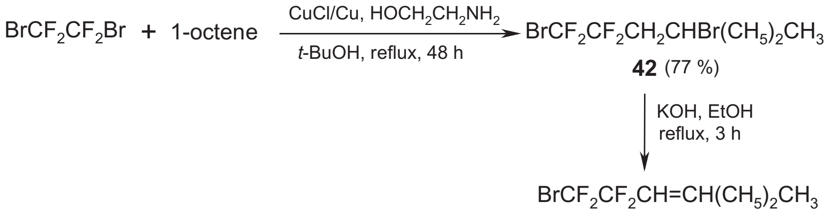
Similarly, quenching of lithiated intermediates of 1,3-disubstituted arene chromium complexes gave regioselectively 2-brominated complexes **41** in high yields [29].



41 (67 - 82 %)

6. Radical addition of $BrCF_2CF_2Br$ to alkenes

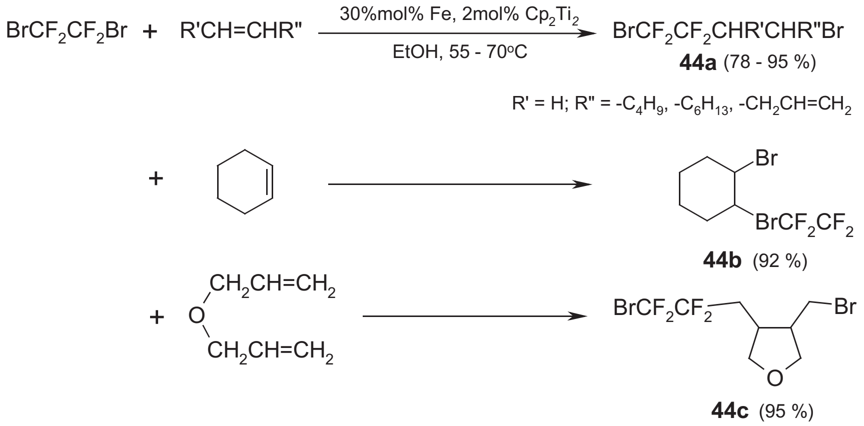
The copper chloride–copper powder–ethanoloamine redox system was used to initiate the addition of a variety of polyhalogenoalkanes, including $BrCF_2CF_2Br$, to 1-octene as a model olefin giving good yields of the 1:1 addition products **42**. Dehydrohalogenation of the adduct **42** gave alkene **43** almost quantitatively [30].



42 (77 %)

43 (95 %)

Addition of Freon 114B2 to alkenes was also successfully promoted by catalytic amount amounts of dichlorobis(π-cyclopentandienyl)titanium (IV) and iron powder affording 1:1 adducts **44a–c** in excellent yields. The reactions were performed in protic solvents (EtOH) without dehalogenation and were assumed to proceed through a radical mechanism [31].



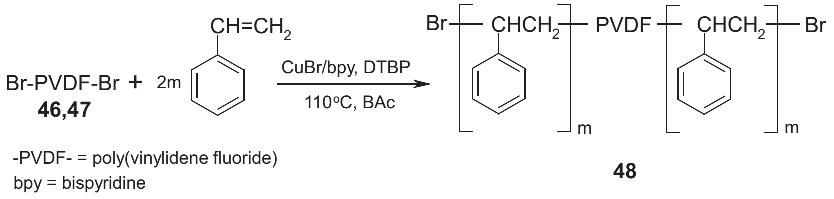
Photoinitiated reactions of $\mathrm{BrCF_2CF_2Br}$ with tetrafluoroethylene using a low-pressure mercury lamp were carried up to give telomers $\mathrm{Br(C_2F_4)_nBr}$ (**45**) with yields of 98, 88 and 54% for $n = 2$, 3 and 4, respectively [32].

$$\mathrm{BrCF_2CF_2Br} + (n-1)\mathrm{CF_2{=}CF_2} \xrightarrow{\mathrm{UV}} \begin{array}{c}\mathrm{Br(CF_2CF_2)_nBr}\\ \mathbf{45}\\ (n = 2\text{–}4,\ \text{up to}\ 94\%)\end{array}$$

Telomerization of vinylidene fluoride using 1,2-dibromotetrafluoroethane as a telogen and various peroxide as radical initiators has been reported [33]. Telomers **46** and **47** with molecular weight of 600–900 units were obtained as liquid, waxy or solid materials containing $\mathrm{CH_2CF_2Br}$, $-\mathrm{CF_2CH_2Br}$ and $-\mathrm{CF_2CF_2Br}$ terminal groups.

$$\mathrm{BrCF_2CF_2Br} + \mathrm{CH_2{=}CF_2} \underset{\mathrm{autoclave}_{60,92\,\mathrm{or}\,147\,^{\circ}\mathrm{C},1\text{–}8\,\mathrm{h}}}{\xrightarrow{\mathrm{peroxide}}} \begin{array}{c}\mathrm{BrCF_2CF_2\text{-}(CH_2CF_2)_n{-}CH_2CF_2Br}\\ +\\ \mathrm{BrCF_2CF_2\text{-}(CH_2CF_2)_n{-}CF_2CH_2Br}\\ \mathbf{46},\mathbf{47}\end{array}$$

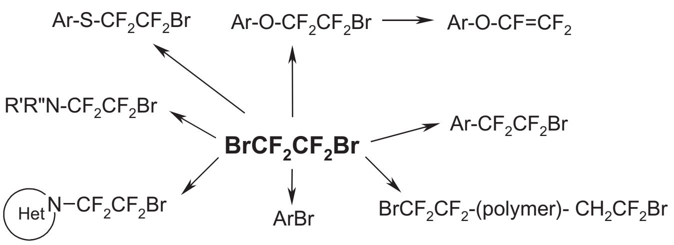
The bromine terminated poly(vinylidene fluorides) of the type **46** and **47** with molecular weight ranging from 2195 to 3870, were used as initiators for polymerization of styrene in the presence of copper(I) bromide and α,α′-bispyridine complexes. Triblock, polystyrene–poly(vinylidene fluoride)–polystyrene, copolymers **48** with molecular weight up to 37,358 units were obtained and characterize [34].



-PVDF- = poly(vinylidene fluoride)
bpy = bispyridine
DTBP = di-*tert*-butylperoxide
BAc = butyl acetate

7. Summary

1,2-Dibromotetrafluoroethane is useful building block for the preparation of numerous fluorine atoms containing compounds like tetrafluoroethyl substituted ethers, thioethers, amines, aromatics, heteroaromatics, alkenes and polymers with terminal bromofluoroalkyl group and other derivatives. Reactions with phenoxides became major synthetic methodology for preparation of aryl trifluorovinyl ethers. The reactions are, in most cases, easy to conduct under mild conditions and usually give good yields of the final products. The key types of compounds, syntheses of which have been reported, are depicted in the scheme below.



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