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Activation of free-radical polyfluoroalkylation of organic substrates with freon $BrCF_2CF_2Br$ using a system of organic base-electron transfer mediator

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ABSTRACT

A polyfluoroalkylation of pyrrole and phenols with freon $BrCF_2CF_2Br$ using sulfur dioxide-substituted pyridine activating system proceeds at the aromatic nucleus by a free-radical mechanism. The essential influence of the basicity of the medium is demonstrated and discussed.

1. Introduction

Polyfluoroalkylation of aromatic and heterocyclic compounds by freons attracted considerably less attention than their fluoroalkylation with iodoperfluoroalkanes, which is largely caused by the lower reactivity of freons as compared to that of perfluoroalkyliodides. However, polyhalogenfluoroalkanes seem interesting as, firstly, frequently cheaper sources of fluoroalkyl groups than iodoperfluoroalakanes and, secondly, as they enable the fluoroalkyl substituent to be further modified by halogen replacement with other groups [1–7].

Interaction of thiophenols, phenols and some heterocyclic compounds with freons is dependent on the state of the substrate (either the starting substrate itself or its salt), on the nature of the freon, and on the reaction conditions. Salts of the above-mentioned compounds mainly undergo halophilic ionic reactions [3,8–13]. For certain freons, in particular CF_2ClBr and CF_2Cl_2, such reactions may proceed by a radical pathway, which suggests a common free radicals-involving step for the halophilic and radical processes [9,10].

The possibility of similar free-radical processes has been shown also for the mild polyfluoroalkylation of thiophenols with freons $BrCF_2CF_2Br$ and $ClCF_2CFCl_2$ [14,15] and of sterically hindered phenols with dibromotetrafluoroethane [16], involving an organic base and an electron transfer mediator. Interestingly, the process feasibility and the products yield have been essentially dependent on the strength of the bases – substituted pyridines – added to the solution [14].

This paper has explored a possibility of employing the above-mentioned "electron transfer mediator-organic base" activating system for polyfluoroalkylation with freons of other classes of organic compounds such as phenols and nitrogen heterocycles, in particular pyrrole. We have payed attention especially to the effect of the strength of the added bases on the fluoroalkylated product yields as one major contributor to the implementation of such processes.

One more interesting *question* is whether the activating system makes it possible to introduce the fluoroalkyl group from the freon into the alkylated substrates' aromatic (heterocyclic) nucleus, rather than to the heteroatom. This would expand the synthetic capability of such processes. For phenols and pyrrol in the presence of an electron transfer mediator and organic bases to promote the radical route of the process, one would expect C-polyfluoroalkylation rather than ionic halophilic O- or N-alkylation [3,8–13]. Such possibility may be related to the phenolates being harder bases than the thiophenolates and consequently less efficient heteroatom-involving free-radical traps. In the case of nitrogen heterocycles, in particular imidazoles, the maximum electronic density [18,19] is mostly concentrated on C- rather than N-atoms, which may result in C-alkylated products.

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C- but not O- or N-alkylated products had been observed earlier in the electrochemical perfluoroalkylation of phenols [17] at fairly low yields and of nitrogen heterocycles [18] with iodoperfluoroalkanes, as well as in the perfluoroalkylation of these compounds, promoted with sodium dithionite [19] and in the presence of metals [20], zinc and sulfur dioxide [21], for which the radical mechanism had been assumed.

2. Results and discussion

Under mild conditions, phenols and pyrrole unlike their salts [3,8–13] do not react with $BrCF_2CF_2Br$. In order to activate electron transfer from these substrates to the freons to form active fluoroalkyl radicals, the electron-donating ability of starting phenols and pyrrole should be enhanced without being converted to salts to avoid halophilic transformations. We have supposed that the electron-donating ability of the substrates could be enhanced by adding strong organic bases to the solution in order to drive the substrate to "the semi-ionised state". To verify this assumption we have added substituted pyridines to solutions of the phenols and pyrrole in aprotic solvents (DMFA, DMSO), as pyridines are capable of forming complexes connected by hydrogen bonds with the above-mentioned substrates [22]. As expected, the electron-donating ability of such complexes significantly increases in comparison with the starting substrates as ascertained by cyclic voltammetry. This indicates a decrease in the oxidation potential of these compounds when adding pyridine; and the higher the pK_a of the added pyridine, the more dramatic the drop will be. For example, Table 1 demonstrates the peak potential (E_p) dependence of electrochemical oxidation of *p*-methylphenol in an acetonitrile-substituted pyridine mixture on the pK_a of the pyridine. In the absence of pyridine for *p*-methylphenol, E_p = 0.91 V.

There is a linear dependence between the E_p of *p*-methylphenol oxidation and the pK_a of the pyridines ($E_p = -0.057$ pK_a + 0.944, r = 0.997). A similar dependence has been observed by us for thiophenols, in particular for *p*-chlorothiophenol $E_p = -0.083$ pK_a + 1.272, r = 0.971) [14]. As the process of electrochemical oxidation of pyrrole in the presence of pyridines is complicated due to the pyrrole polymerisation on the electrode to form a passivating polymer film, therefore it has been rather difficult to determine the precise values of E_p for the pyrrole.

Enhancing the donor strength of phenols and pyrrole via addition of the pyridines to the aprotic solvent is a necessary but insufficient condition for the substrate-to-freon electron transfer and the progress of fluoroalkylation. To further activate the process we have added sulfur dioxide as an effective electron transfer mediator, which we have used previously for the fluoroalkylation of some organic substrates with freons [14–16]. The ability of SO_2 to accept an electron from phenol or pyrrole complexes with nitrogen bases has been confirmed by the results of our spectrophotometric investigations: addition of sulfur dioxide to a solution of pyrrole with β-picoline in a deaerated DMSO causes a blue colouring of the solution, and concurrently in the electron spectrum we observe an absorption band (580 nm), attributed to an $S_2O_4^{-\bullet}$ anion radical, i.e. dimerisation product of the $SO_2^{-\bullet}$ and starting sulfur dioxide [23]. Importantly, the $S_2O_4^{-\bullet}$ anion radical absorption band appears solely in the simultaneous presence of pyrrole and β-picoline, whereas without β-picoline there is no sulfur dioxide reduction with pyrrole to form $S_2O_4^{-\bullet}$. By adding the freon to the solution under investigation, the absorption band of the $S_2O_4^{-\bullet}$ anion radical rapidly disappears, which may be relevant to its interaction with the freon. Evidence for subsequent electron transfer from $SO_2^{-\bullet}$ anion radical to freon can be provided by the results of our previous electrochemical investigations into cathodic SO_2 reduction in the presence of the freons [14–16], which demonstrates significant catalytic currents in SO_2 cyclic voltammograms when adding freon $BrCF_2CF_2Br$ to the solution.

Table 1
Peak potential (E_p) dependence of electrochemical oxidation of *p*-methylphenol on the pK_a of the pyridines added to the system [*p*-$CH_3C_6H_4OH$] = 0.001 M; [pyridine] = 2.5×10^{-3} M; Pt; $NaClO_4$ 0.1N; CH_3CN, Ag/Ag^+ 25 °C.

	2-Chloropyridine	Pyridine	β-Picoline	2,4-Lutidine	γ-Collidine
pK_a	0.72	5.23	5.97	6.82	7.60
E_p (V)	0.90	0.66	0.60	0.55	0.51

Table 2
Peak potential dependence of electrochemical reduction of sulfur dioxide on the pK_a of the pyridines added to the solution [SO_2] = 7×10^{-5} mol; [pyridine] = 2.5×10^{-3} mol; DMSO; Pt; Bu_4NClO_4 0.1N; Ag/Ag+; 25 °C.

	2-Acetylpyridine	Pyridine	2,5-Lutidine	γ-Collidine
pK_a	3.18	5.23	6.25	7.60
E_p, V	−1.34	−1.16	−0.98	−0.88

Nitrogen-containing heterocyclic bases critically influence not only the electron-donating properties of alkylated substrates but also the ability of the SO_2 mediator to accept an electron from the substrate–pyridines complexes. The last fact could also be an important reason for the influence of the basicity of the medium on the efficiency of fluoroalkylation processes using the "electron transfer mediator-base" activating system. As follows from Table 2, increasing basicity of the pyridines, added to the solution, markedly increases the electron-accepting capacity of the sulfur dioxide too, which, for its part, should facilitate the freon activation process.

We have elucidated the ability of our activating system to activate fluoroalkylation of nitrogen-containing nucleophiles, pyrrole in particular. We have ascertained that the reaction of pyrrole with $BrCF_2CF_2Br$ in aprotic solvents in the presence of sulfur dioxide and pyridine leads to pyrrole fluoroalkylation to form 2-(2-bromotetrafluoroethyl)pyrrole by a procedure similar to alkylation of thiophenols with this freon [14]. The yields of the fluoroalkylated pyrroles depend significantly on the sulfur dioxide concentration (Table 3) and on the pK_a of the added base (Table 4).

Adding pyridines such as 2-chloropyridine or 2-acetylpyridine to the solution would not ensure the pyrrole's sufficient electron-donating capacity for freon activation and the fluoroalkylation process fails. With further increasing pK_a of pyridines, the fluoroalkylation process proceeds more efficiently and the yield of 2-(2-bromotetrafluoroethyl)pyrrole becomes practically quantitative when adding 2,5-lutidine. In the case of γ-collidine the desired product yield drops, which obviously accounts for the debromination of the freon with formation of tetrafluoroethylene,

Table 3
Product yields of pyrrole–$BrCF_2CF_2Br$ reaction in the presence of SO_2 [pyrrole] = 1×10^{-3} mol; DMSO; 35 °C.

[SO_2] ($\times 10^{-3}$ mol)	[β-picoline] ($\times 10^{-2}$ mol)	[$BrCF_2CF_2Br$] ($\times 10^{-3}$ mol)	Product yields (%)
–	1.0	4.0	–
0.5	1.0	4.0	18
1.0	1.0	4.0	51
2.0	1.0	4.0	85
4.0	1.0	4.0	55
2.0	1.0	4.0	–[a]
1.0	1.0	1.0	78
1.0	–	4.0	–

[a] *p*-Dinitrobenzene is added.

Table 4
Dependence of the yield of the polyfluoroalkylated pyrrole on the basicity of the pyridines added to the system [pyrrole] = 0.5×10^{-3} mol; [SO_2] = 0.44×10^{-3} mol; [$BrCF_2CF_2Br$] = 0.8×10^{-3} mol; [substituted pyridine] = 1.0×10^{-2} mol; DMSO, 35 °C.

Base	pK_a	Product yield (%)	CF_2=CF_2
2-Chloropyridine	0.72	–	–
2-Acetylpyridine	3.18	–	–
Pyridine	5.23	78	–
β-Picoline	5.97	89	–
2,5-Lutidine	6.25	95	–
γ-Collidine	7.60	62	8
β-Picoline[a]	5.97	–	–

[a] *p*-Dinitrobenzene is added.

Table 5
Dependence of the yield of the polyfluoroalkylated *p*-methylthiophenol on the basicity of the pyridines added to the system. [*p*-methylthiophenol] = 5×10^{-4} mol; [$BrCF_2CF_2Br$] = 1×10^{-3} mol; [SO_2] = 7×10^{-4} mol; DMFA; 25 °C.

Base	pK_a	Yield of *p*-$CH_3C_6H_4SCF_2CF_2Br$ (%)
2-Chloropyridine	0.72	–
2-Acetylpyridine	3.18	–
Pyridine	5.23	78
2,5-Lutidine	6.25	98
γ-Collidine	7.60	99
Pyridine[a]	5.23	28

[a] *p*-Dinitrobenzene is added.

and presumably for the precipitation of sulfur dioxide–γ-collidine salt.

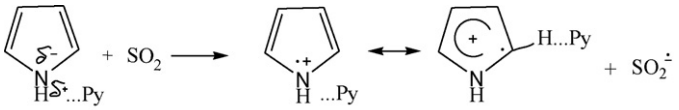
Similarly, the significant influence of the pyridine basicity on the fluoroalkylation process using the "sulfur dioxide-substituted pyridine" activating system has been observed by us for fluoroalkylation of thiophenols with $BrCF_2CF_2Br$ (Table 5).

As follows from Tables 4 and 5, for the pyrrole and thiophenol alike the maximum yields of fluoroalkylation product are obtained in the range of pK_a = 5–6 for pyridines used to raise the electron-donating capacity of the substrates.

In the case of fluoroalkylation of both pyrrole (Table 4) and thiophenols (Table 5) addition of *p*-dinitrobenzene as a free-radicals trap to the reaction mixture sharply inhibits the formation of the desired products, which may support the hypothesis that these processes proceed by a free-radical route.

Last but not least the radical nature of the processes involving the "electron transfer mediator-base" activating system is supported by the observation that the fluoroalkylation of thiophenol with $ClCF_2CFCl_2$ gave thioethers *p*-$XC_6H_4SCFClCF_2Cl$ (X = CH_3, H, Cl) [15], rather than the alkylation products of thiophenol potassium salt, *p*-$XC_6H_4SCF_2CFCl_2$, formed by a halophilic mechanism [10].

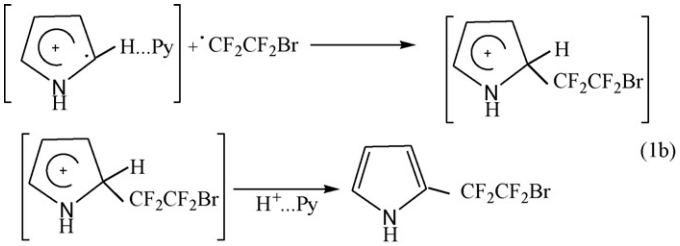
The probable mechanism of pyrrole fluoroalkylation with $BrCF_2CF_2Br$ in the presence of pyridines and sulfur dioxide can be represented by Scheme 1a and 1b.



(1a)

$$SO_2^{\bullet-} + BrCF_2CF_2Br \longrightarrow SO_2 + {}^{\bullet}CF_2CF_2Br + Br^-$$

The way in which the process unfolds further is not so clear. The nascent active radicals $^{\bullet}CF_2CF_2Br$ may then react with the pyrrole cation radical (its complex with substituted pyridines), which is accompanied by proton abstraction to form 2-(2-bromotetrafluoroethyl)-pyrrole:



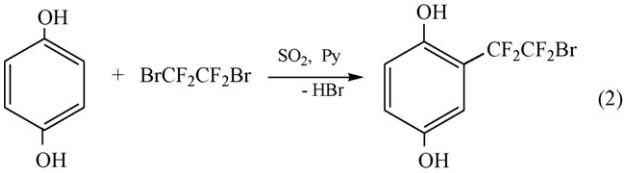
(1b)

Both the bromide ion and the pyridine may act as the proton acceptor.

As seen from the above-mentioned mechanism, the base is very important not only in the pyrrole oxidation step (Scheme 1a), enhancing the ability of the pyrrole to donate the electron to the mediator and then to the freon, but also in the proton abstraction step (1b). With increasing pK_a of the base, its proton-accepting ability is increasing, too.

However, it cannot be ruled out that the fluoroalkylation process may proceed through a radical-nucleophilic mechanism $S_{RN}1$ [24–27] that, as follows from Refs. [1,3,18], may lead to C-fluoroalkylated nitrogen-containing heterocycles.

We studied the possibility of mild fluoroalkylation of phenols with freon $BrCF_2CF_2Br$ using the above-mentioned activating system. As pointed out above, the fluoroalkyl radical attack was expected to proceed at the *o*- or *p*-positions of the aromatic ring forming fluoroalkylated phenols rather than by phenolate fluoroalkylation yielding ethers [10–12,28]. Using hydroquinone as one of the most active electron donors among the phenols, we explored a phenols–$BrCF_2CF_2Br$ reaction in the presence of SO_2 as the electron transfer mediator and of β-picoline used to enhance the electron-donating capacity of the fluoroalkylated substrates. The process was run at 35 °C for 3–4 h in sealed ampoules by adding sulfur dioxide to a solution containing hydroquinone, freon and β-picoline in oxygen-free organic solvents, i.e. dimethylformamide or dimethylsulphoxide. It has been ascertained that using the "sulfur dioxide-substituted pyridine" activating system, under the conditions given above, hydroquinone can be polyfluoroalkylated in the aromatic ring with $BrCF_2CF_2Br$ (Scheme 2):



(2)

The yield of the polyfluoroalkylated hydroquinone in the presence of equimolar amounts of sulfur dioxide and excess β-picoline in dimethylformamide at room temperature stands at 66% and gets practically quantitative (92%) at twofold SO_2 excess. Addition of *p*-dinitrobenzene as a radical trap markedly inhibited the process, which again supports its radical nature.

The "sulfur dioxide-substituted pyridine" activation system helps to introduce $BrCF_2CF_2$-group of freon not only to the aromatic ring of hydroquinone, but also into phenol to form *para*- and *ortho*-fluoroalkylated products with a combined yield of 51% at 35 °C. These results will be subject of our next publication.

3. Conclusion

Thus, a system of an organic base (substituted pyridines) and an electron transfer mediator (sulfur dioxide) enables mild and efficient polyfluoroalkylation of the aromatic ring of phenols and pyrrole with 1,2-dibromotetrafluoroethane. Complexation of substituted pyridines with phenols and pyrrole enhances the electron-donating properties of the substrates, activates freon

using a mediator to form active radicals and makes the process proceeding by a radical route.

4. Experimental

4.1. General

Melting points were uncorrected. ^{1}H NMR and ^{19}F NMR spectra were recorded in δ (ppm) using a Bruker-CXP-90 (90 MHz) spectrometer (DMSO-d_6, vs. TMS (^{1}H) and CCl_3F (^{19}F) as internal standard), the ^{13}C NMR spectra using a Bruker AVANCE 400 spectrometer ($CDCl_3$ vs. TMS). The elemental analysis was carried out on elemental analyzer "Carlo Erba", model 1106. Cyclic voltammetry experiments were carried out in the undivided three electrode analytical cell; working electrode: disk cathode of platinum; auxiliary electrode: platinum wire; reference electrode: Ag/AgCl. The solvent of analytical grade (10 mL) contained supporting salt (0.1 M Bu_4NBF_4). The cell was purged for 15 min with nitrogen. The research was carried out using EP 20A potentiostate- and PC-based computerized electrochemical facilities. The yields of polyfluoroalkylated products (Tables 3–5) were calculated from ^{19}F NMR spectra respectively to the pyrrole and hydroquinone.

4.1.1. Reaction of pyrrole with $BrCF_2CF_2Br$ in the presence of sulfur dioxide and β-picoline

1,2-Dibromotetrafluoroethane (0.48 mL, 4.0×10^{-3} mol) and sulfur dioxide (SO_2 solution in DMFA, 2×10^{-3} mol) were added to a solution of pyrrole (0.067 g, 1×10^{-3} mol) in a mixture of DMFA (2.5 mL) with β-picoline (0.4 mL, 4.0×10^{-3} mol) under an argon atmosphere. The sealed ampoule was kept at 35 °C for 4–5 h. The reaction mixture was diluted with 30 ml of 17% solution of HCl, three times extracted with hexane or chloroform, organic layer washed with water, dried and solvent distilled. The product 2-(2-bromotetrafluoroethyl)pyrrole was distilled under reduced pressure. b.p. 66–68 °C (15 Torr); yield 82% (based on pyrrole); ^{1}H NMR δ: 6.2 (s, 1H (4)); 6.5 (s, 1H (3)); 7.0 (s, 1H (5)); 11.9 (s, 1H, N–H); ^{19}F NMR δ: 65.7 (t, 2F, CF_2, J_{FF} = 5.6 Hz); 102,8 (t, 2F, CF_2Br, J = 5.6 Hz). Anal. Calcd. for $C_6H_4BrF_4N$: C, 29.3; H, 1.6; N, 5.7. Found: C, 29.5; H, 1.7; N, 5.8.

4.1.2. Reaction of pyrrole with $BrCF_2CF_2Br$ in the presence of sulfur dioxide, β-picoline and p-dinitrobenzene

The test was carried out similarly to the above-mentioned protocol (Section 4.1.1) however, *p*-dinitrobenzene (0.0336 g, 2×10^{-4} mol) was added to the reaction mixture. Further treatment of reaction products and their determining were carried out similarly to the above mentioned. Products containing fluorine were not revealed.

4.1.3. Reaction of pyrrole with $BrCF_2CF_2Br$ in the presence of sulfur dioxide and different substituted pyridines

The tests were carried out similarly to the above-mentioned protocol (Section 4.1.1), however, different substituted pyridines (2×10^{-4} mol) mentioned in Table 4 were added instead of β-picoline to the reaction mixture. Further treatment of reaction products and their determining were carried out similarly to the above mentioned.

4.1.4. Reaction of hydroquinone with $BrCF_2CF_2Br$ in the presence of sulfur dioxide and β-picoline

1,2-Dibromotetrafluoroethane (0.2 mL (2.0×10^{-3} mol) and sulfur dioxide (0.17 mL of SO_2 solution in DMFA, 5×10^{-4} mol SO_2) were added to a solution of hydroquinone (0.055 g, 5×10^{-4} mol) in a mixture of DMFA (2.5 mL) with β-picoline (0.2 mL, 2.0×10^{-3} mol) under an argon atmosphere. The sealed ampoule was kept at 35 °C for 3–4 h. The reaction mixture was diluted with 30 ml of 17% solution of HCl, three times extracted with hexane or chloroform, organic layer washed with water, dried and solvent distilled. The reaction product *p*-HO-$C_6H_3(CF_2CF_2Br)$-OH was isolated by silica gel column chromatography with ether–dichloroethane (1:1) as eluent; yield 85% respectively to the hydroquinone; colorless crystals, m.p. 76 °C. ^{1}H NMR δ: 6.5–6.8 (m, 3H, C_6H_3); ^{19}F NMR δ: 68.5 (t, 2F, CF_2, J_{FF} = 4.8 Hz), 109.7 (t, 2F, CF_2Br, J_{FF} = 4.8 Hz); Anal. Calcd. for $C_6H_5BrF_4O_2$: C, 33.2; H, 1.7. Found: C, 33.2; H, 1.7.

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