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Electrolytic partial fluorination of organic compounds. Part 29¹. Anodic mono- and difluorination of 2-benzoxazolyl sulfides

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Abstract

Anodic fluorination of 2-benzoxazolyl sulfides using various fluoride salts and dimethoxyethane as a supporting electrolyte and a solvent, respectively, resulted in the formation of the corresponding mono- and difluorinated products in reasonable yields. The monofluorination took place selectively at the position α to the sulfur atom while the difluorination resulted at both the α -position and the benzene ring while the expected α , α -difluorination did not take place at all. Moreover, anodic fluorination of the electrolytic solvent, DME, which has never been reported so far, was also observed as a side-reaction and this fluorination was further confirmed by carrying out anodic fluorination of DME itself in the absence of a substrate sulfide. \bigcirc 1999 Elsevier Science S.A. All rights reserved.

Keywords: Selective anodic fluorination; Benzoxazolyl sulfides; Fluoride salt; Dimethoxyethane

1. Introduction

Benzoxazole derivatives are well-known to exhibit antibacterial [2], antimicrobial [3], antifungal [4], antiinflammatory and analgesic [5,6] activities. On the other hand, fluoroorganic compounds have attracted a great deal of interest due to their considerable importance as medicines and agrochemicals [7-10]. Therefore, introduction of fluorine atom into the benzoxazoles 2a-d or their side chains may enhance their biological activities. However, direct fluorination is not always straightforward and very often requires hazardous or costly fluorinating agents. Recently, anodic fluorination proved to be more convenient because of its selectivity and safety [11-22]. For example, anodic fluorination of some heterocyclic sulfides bearing electron-withdrawing groups at their α -position was firstly explored by our research group and the fluorine atom has been introduced selectively to the α -position but the heterocyclic ring fluorination did not take place at all [23-29].

In our continuous studies on the anodic fluorination of a variety of heterocyclic sulfides [23–29], we disclose herein the anodic behavior of 2-mercaptobenzoxazoles 2a-d

toward various fluoride salts and electrolytic solvents. Anodic monofluorination at α -position to the sulfur atom took place preferentially and by-products difluorinated at both a side-chain and a benzene ring were also formed. Interestingly, anodic fluorination of 1,2-dimethoxyethane (DME), solvent used for the electrolysis, was also observed.

2. Results and discussion

2.1. Preparation of 2-benzoxazolyl sulfides 2a-c

Treatment of 2-mercaptobenzoxazole (1) with α -chloroacetone, ethyl α -chloroacetate, and α -chloroacetonitrile in refluxing THF, in the presence of K₂CO₃, afforded the corresponding 2-benzoxazolyl sulfides **2a–c**, respectively, in good yields, as shown in Scheme 1.

2.2. Oxidation potentials of 2-benzoxazolyl sulfides

The oxidation potentials of 2-benzoxazolyl sulfides 2a-dand the benzoxazole (6) were measured by cyclic voltammetry in an anhydrous acetonitrile solution containing Bu₄Nm·BF₄ (0.1 M) using a divided cell and platinum electrodes. All benzoxazoles 2a-d and 6 displayed irreversible oxidation peaks. Among all compounds under study, the benzoxazole (6) has the highest oxidation potential (2.32 V) and 2-methylthiobenzoxazole (2d) has the lowest

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 Table 1

 Oxidation potentials of 2-benzoxazolyl sulfides

O S EWG

Sulfide		$E_{\rm p}^{\rm ox}$ (V vs. SSCE) ^a
No	EWG	
2a	COCH ₃	2.08
2b	COOEt	1.96
2c	CN	2.22
2d	Н	1.90
Benzoxazole		2.32

^aIn 0.1 M Bu₄N·BF₄/MeCN. Anode: Pt plate. Sweep rate: 100 mVs⁻¹.

one (1.90 V). Substitution of an electron-withdrawing group at the position α to the sulfur atom increases the oxidation potentials in the order COOEt<COCH₃<CN as shown in Table 1.

2.3. Current-potential curves

Before carrying out the anodic fluorination, the currentpotential curves for acetonitrile and DME in the presence and absence of 2a in 0.3 M Et₄NF·4HF were measured in order to select the proper electrolytic solvent. As shown in Fig. 1(B), addition of 2-acetonylmercaptobenzoxazole (2a) to the DME electrolytic solution led to an anodic shift. This indicated that both 2a and DME should be oxidized simultaneously. Therefore, this electrolytic system does not seem to be preferable for anodic fluorination. On the other hand, a large cathodic shift was observed after addition of 2a to the electrolytic acetonitrile solution as shown in Fig. 1(A). Compared with the former case, the latter electrolytic solution gave a much larger faradaic current due to the discharge of 2a. Therefore, acetonitrile seemed to be suitable for anodic fluorination. Experimental results showed however, the reverse trend: DME was found to be a suitable electrolytic solvent.

2.4. Anodic fluorination of 2-S-substituted mercaptobenzoxazoles **2a–d**

Anodic fluorination of 2-acetonylmercaptobenzoxazole (2a), as a model example, was studied in details under various conditions and the results are summarized in Table 2.

Instead of selective fluorination at the α -position to the electron-withdrawing group as reported, previously by us for the cases of 2-pyridyl [24,27] and 2-benzothiazolyl sulfides [26], the 2-benzoxazolyl sulfide (**2a**) differed to some extent and the fluorination took place at the position α to the sulfur atom as well as at the benzene ring as depicted in Scheme 2.

When the reaction was monitored by GC–MS during the electrolysis, it was found that fluorination took place firstly at the α -position till 5 F mol⁻¹, then diffuorination started to take place at the benzene ring. However, the expected α , α -diffuorinated product **5a**, like the case of the corresponding phenyl sulfides [21], was not formed at all during the electrolysis. Among the fluoride salts used, Et₄NF·4HF was the best fluorinating agent in DME and the α -fluorinated product **3a** was obtained in reasonable yield as a major product. Regardless of supporting electrolytes, a small amount of diffuorinated product **4a** was obtained as a by-product when DME was used as a solvent (Scheme 2). The position of the additional fluorination could not be deter-



Fig. 1. Current–potential curves of the electrolytic solutions: (A) 0.3 M Et₄NF·4HF/MeCN (20 ml) and (B) 0.3 M Et₄NF·4HF/DME (20 ml), in the presence (\blacktriangle) and the absence (\square) of substrate **2a** (1 mmol).

	• •					
Run	Supporting electrolyte	Solvent	Charge passed (F/mol) ^a	Yield (%) ^b		
				3a	4 a	Others
1	Et ₄ NF·4HF	DME	9.5	17	3	+ ^{c,e}
2	Et ₄ NF·4HF	DME	18	39	8	$+^{c}$
3	Et ₄ NF·3HF	DME	7	10	2	$+^{c,e}$
4	Et ₄ NF·3HF	DME	18	34	7	$+^{c}$
5	Et ₃ N·3HF	DME	18	35	7	$+^{c}$
6	Et ₄ NF·4HF	MeCN	7	0	0	$+^{d}$
7	Et ₃ NF·3HF	MeCN	7	2	0	$+^{d}$
8	Et ₄ NF·4HF	CH ₂ Cl ₂	8	2	0	$+^{e}$
9	Et ₄ NF·4HF	(Neat)	8	<1	0	$+^{e}$

 Table 2

 Anodic fluorination of 2-acetonylmercaptobenzoxazole (2a)

^aConstant current electrolysis (6 mA cm⁻²). Anode and cathode were Pt plates (3×2 cm²).

^bDetermined by ¹⁹F-NMR spectra.

^cCH₃OCH₂CH₂OCH₂F was formed.

^dUnidentified solid product was obtained.

eStarting material was recovered.





mined due to its extremely low yield, however, it seems to be the 5- or 6-position of the benzoxazole ring like the case of benzoxazole ($\mathbf{6}$) (Scheme 7).

On the other hand, acetonitrile and dichloromethane were not suitable as a solvent even when $Et_4NF \cdot 4HF$ was used as a supporting electrolyte. Thus, DME gave much better results than acetonitrile, which cannot be explained from *I*-*E* curves of **2a** and the solvents, DME and acetonitrile. This can be explained in terms of the ability of DME to solvate the cationic part of the fluoride salts, due to its higher donor number (23.9) than acetonitrile (14), leaving a naked fluoride anion which can consequently attack easily the anodically generated cationic intermediate of the substrate. A plausible mechanism is outlined in Scheme 3.

Although $Et_4NF.4HF/DME$ was effective for the anodic fluorination, $Et_4NF.4HF$ without any solvent was not effective at all (run 9). This is quite different from the anodic fluorination of arenes using neat $Et_4NF.4HF$ [30].

It is notable that anodic fluorination of DME (Scheme 4) was observed as a side reaction, in all cases using DME as an electrolytic solvent, which may reflect the poor current efficiency in runs 1–5.

Fluorinated DME was established based on ¹⁹F NMR and mass spectrum of the reaction product. A triplet signal $(J_{\rm HF}=56 \text{ Hz})$ was observed at δ -74.61 ppm in the ¹⁹F NMR and molecular ion peak of monofluorinated DME, *m/e* 108 was also detected by the mass spectrum although the molecular ion peak was rather weak. The formation of monofluorinated DME was further confirmed by carrying out the anodic fluorination of DME itself without substrate **2a** and the same results were obtained. A possible mechanism is depicted in Scheme 5.





Next, we extended the anodic fluorination to other 2substituted mercaptobenzoxazoles **2b–d** using DME as an electrolytic solvent (Scheme 6). As shown in Table 3, mono- and difluorinated products **3b,c** and **4b,c** were obtained in reasonable yields, respectively, regardless of



the electron-withdrawing groups. In contrast to these cases, 2-methylthiobenzoxazole (2d) devoid of an electron-withdrawing group gave an extremely low yield of fluorinated products 3d and 4d. The fluorination of DME was observed in all cases and particularly in the case of 2d, the difluorination of DME took place considerably. However the α , α difluorinated products were not detected at all regardless of sulfides.

Anodic fluorination of benzoxazole (6) was also performed. The formation of two monofluorinated products 7 and 8 were detected and confirmed by ¹⁹F NMR [δ –38.02 (ddd, J=8.27, 4.60, 3.67 Hz) and –48.18 (dd, J=8.25, 4.60 Hz)] and GC–MS spectra [m/z 137 (M⁺), 110 (M⁺– HCN)], respectively, although their yields were extremely low. In this case, fluorination took place at the benzene ring but the oxazole ring was not fluorinated at all (Scheme 7). This trend is quite similar to the anodic fluorination of benzothiazole [26] and oxindole [25].

N-Fluoropyridinium salts are known to be a good fluorinating reagents [31,32]. Therefore, the chemical fluorination of compound 2a, as a model compound, was also attempted. However, treatment of 2a with various *N*-fluoropyridinium triflates 9a-c in dichloromethane at either room temperature or under refluxing resulted in no formation of any desired fluorinated products as shown in Scheme 8. Therefore, the electrochemical fluorination proved to be superior to the conventional chemical method.



3. Experimental

3.1. General

Caution. Et₃N·3HF and Et₄NF·*n*HF (n=3,4) are toxic and if in contact with skin cause a serious burn, so proper safety precautions should be taken at all times. It is therefore recommended to protect the hands with rubber gloves.

¹H NMR, ¹⁹F NMR and ¹³C NMR spectra were recorded at 270, 254 and 68 MHz, respectively, in CDCl₃ as a solvent using a JEOL EX-270 spectrometer. The chemical shifts for ¹H and ¹³C NMR are given in δ ppm downfield from internal TMS and ¹⁹F NMR are given in δ ppm downfield from external perfluorobenzene. Mass spectra were obtained with Shimadzu GCMS-QP2000A and JEOL JMS-700 mass spectrometers. Cyclic voltammetry was performed using a Hokuto Denko Potentiostat/Galvanostat HA 6-151 at a scan speed of 100 mV s⁻¹ in 0.1 M Bu₄N·BF₄/CH₃CN. Preparative electrolysis experiments were carried out using a Metronix Corp. (Tokyo) constant-current power supply.



Table 3	
Anodic fluorination of 2-benzoxazolyl sulfides 2a–d .	

Sulfide	EWG	Supporting electrolyte	Charge passed (F/mol) ^a	Yield (%) ^b		
				3	4	Others ^c
2a	COCH ₃	Et ₄ NF·4HF	18	3a (39)	4a (8)	+
2b	COOEt	$Et_4NF \cdot 4HF$	10	3b (35)	4b (13)	+
2b	COOEt	Et ₃ N·3HF	10	3b (29)	4b (8)	+
2c	CN	Et ₄ NF·4HF	18	3c (17)	4c (6)	+
2c	CN	Et ₃ N·3HF	18	3c (12)	4c (8)	+
2d	Н	Et ₄ NF·4HF	18	3d (2.2)	4d (2)	++

^aConstant current electrolysis (6 mA cm⁻²). Anode and cathode were Pt plates (3×2 cm²).

^bDetermined by ¹⁹F-NMR spectra.

^cCH₃OCH₂CH₂OCH₂F was formed.

3.2. Preparation of 2-S-substituted mercaptobenzoxazoles 2a-c: general procedure

To a stirred solution of 2-mercaptobenzoxazole (1) (3.02 g, 20 mmol) in THF (40 ml), in the presence of potassium carbonate (3.4 g, 25 mmol), was added the appropriate α -chloro compound (20 mmol). The reaction mixture was heated under reflux for 1 h, then left to cool. The inorganic salts were filtered off and the filtrate was evaporated under vacuum. The remaining product was crystallized from hexane/isopropanol (5:1) or methanol to give the desired products **2a–c**.

1-(2-Benzoxazolylthio)-2-propanone (2a) [33]: 78% yield, m.p. 68–69°C; ¹HNMR δ 2.40 (s, 3H), 4.23 (s, 2H), 7.27 (m, 2H), 7.44 (dt, *J*=7.26, 1.65 Hz, 1H), 7.57 (dt, *J*=6.93, 1.65 Hz, 1H); MS *m/e* 207 (M⁺), 164 (M⁺– COCH₃); Anal. Calcd. for C₁₀H₉NO₂S: C, 57.96%; H, 4.38%; N, 6.76%. Found C, 58.14%; H, 4.22%; N, 6.53%.

Ethyl α-(2-*benzoxazolylthio*)*acetate* (**2b**) [34]: 78% yield, m.p. 46°C; ¹H NMR δ 1.29 (t, *J*=7.26 Hz, 3H), 4.12 (s, 2H), 4.24 (q, *J*=7.26 Hz, 2H), 7.28 (m, 2H), 7.44 (dt, *J*=5.94, 1.65 Hz, 1H), 7.60 (dt, *J*=5.94, 1.65 Hz, 1H); MS *m/e* 237 (M⁺), 164 (M⁺–COOEt); Anal. Calcd. for C₁₁H₁₁NO₃S: C, 55.68%; H, 4.67%; N, 5.90%. Found C, 55.78%; H, 4.59%; N, 5.78%.

α-(2-Benzoxazolylthio)acetonitrile (**2c**) [35]: 84% yield, m.p. 95°C; ¹HNMR δ 4.11 (s, 2H), 7.34 (m, 2H), 7.48 (m, 1H), 7.66 (m, 1H); MS *m/e* 190 (M⁺), 164 (M⁺–HCN), 150 (M⁺–CH₂CN); Anal. Calcd. for C₉H₆N₂OS: C, 56.83%; H, 3.18%; N, 14.73%. Found C, 56.84%; H, 2.99%; N, 14.81%.

3.3. Anodic fluorination of 1-(2-benzoxazolylthio)-2propanone (2a)

Electrolysis was carried out with platinum electrodes $(3 \times 2 \text{ cm}^2)$ in 0.3 M Et₄NF·4HF/DME (15 ml) to which the substrate 2a (1 mmol) was added, using an undivided cell under nitrogen atmosphere at ambient temperature. Constant current (6 mA cm⁻²) was applied until the starting material 2a was almost consumed. The course of the reaction was monitored by TLC and GC-MS. After complete electrolysis, the electrolytic solution was neutralized with 10% NaHCO₃ solution followed by extraction with ether several times. The combined extracts were dried over anhydrous MgSO₄, then evaporated under vacuo and the product yields were estimated by ¹⁹FNMR spectroscopy. The solvent was removed by evaporation and the oily residue was purified by passing through column chromatography on silica gel using hexane/ethyl acetate (5:1) as an eluent.

This typical electrolysis procedure was also carried out for the other benzoxazoles 2b-d and 6 as well as DME.

1-(2-Benzoxazolylthio)-1-fluoro-2-propanone (3a): ¹H NMR δ 2.50 (d, J=3.63 Hz, 3H), 6.81 (d, J=50.48 Hz, 1H), 7.33 (m, 2H), 7.49 (m, 1H), 7.65 (m, 1H); ¹⁹F NMR δ -85.89 (dq, J=50.55, 3.68 Hz); MS *m/e* 225 (M⁺), 183 $(M^+-CH_2=C=O)$, 163 (M^+-CH_3COF) , 150 $(M^+-CHFCOCH_3)$; HRMS: Calcd. for $C_{10}H_8FNO_2S$: *m/e* 225.0260; Found 225.0263.

Ethyl α-(2-*benzoxazolylthio*)-α-*fluoroacetate* (**3b**): ¹HNMR δ 1.35 (t, *J*=7.26 Hz, 3H), 4.37 (q, *J*=7.26 Hz, 2H), 6.94 (d, *J*=50.56 Hz, 1H), 7.33 (m, 2H), 7.50 (m, 1H), 7.67 (m, 1H); ¹³C NMR δ 13.90, 63.31, 91.66 (dd, *J*=238, 6.1 Hz), 110.29, 119.30, 124.88 (d, *J*=9.5 Hz), 141.33, 152.16, 159.15 (d, *J*=2.4 Hz), 164.48 (d, *J*=25.7 Hz); ¹⁹F NMR δ -85.52 (d, *J*=50.56 Hz); MS *m/e* 255 (M⁺), 182 (M⁺-COOEt), 150 (M⁺-CHFCOOEt); HRMS: Calcd. for C₁₁H₁₀FNO₃S: *m/e* 255.0365; Found 255.0364. Anal. Calcd. for C₁₁H₁₀FNO₃S: C, 51.76%; H, 3.95%; N, 5.49%. Found C, 51.75%; H, 3.82%; N, 5.30%.

α-(2-Benzoxazolylthio)-α-fluoroacetonitrile (**3c**): ¹HNMR δ 7.22 (d, J=49.16 Hz, 1H), 7.37 (m, 2H), 7.53(m, 1H), 7.71 (m, 1H); ¹⁹FNMR δ -81.13 (d, J=48.72 Hz); MS *m/e* 208 (M⁺), 191 (M⁺-HCN), 150 (M⁺-CHFCN); HRMS Calcd. for C₉H₅FN₂OS: *m/e* 208.0106; Found 208.0114.

 $\begin{array}{l} 1\mbox{-}[5(6)\mbox{-}Fluoro\mbox{-}2\mbox{-}benzoxazolylthio\mbox{]}\mbox{-}1\mbox{-}fluoro\mbox{-}2\mbox{-}propanone (4a): 1H NMR δ 2.51 (d, $J\mbox{=}3.62 Hz, 3H), $6.76 (d, $J\mbox{=}50.48 Hz, 1H), $7.10 (m, 1H), $7.22 (dd, $J\mbox{=}7.91, $2.64 Hz, 1H), $7.58 (dd, $J\mbox{=}8.91, $4.95 Hz, 1H); 19F NMR δ -38.29 (m, 1F), $-85.89 (dq, $J\mbox{=}50.55, $3.68 Hz, 1F); MS m/e 243 (M^+), $201 (M^+\mbox{-}CH_2\mbox{=}C\mbox{=}0), $168 (M^+\mbox{-}CHFCOCH_3);$HRMS: Calcd. for $C_{10}H_7F_2NO_2S$, m/e 243.0165; Found 243.0165. \end{array}$

Ethyl α-[5(6)-*Fluoro-2-Benzoxazolylthio*]-α-*fluoroacetate* (**4b**): ¹H NMR δ 1.35 (t, *J*=7.26 Hz, 3H), 4.37 (q, *J*=7.26 Hz, 2H), 6.88 (d, *J*=50.48 Hz, 1H), 7.09 (m, 1H), 7.24 (dd, *J*=7.91, 2.31 Hz, 1H), 7.61 (dd, *J*=8.91, 4.95 Hz, 1H); ¹⁹F NMR δ -38.23 (m, 1F), -85.50 (d, *J*=50.56 Hz, 1F); MS *m/e* 273 (M⁺), 200 (M⁺-COOEt), 168 (M⁺-CHFCOOEt); HRMS Calcd. for C₁₁H₉F₂NO₃S: *m/e* 273.0271; Found 273.0269.

α-[5(6)-Fluoro-2-Benzoxazolylthio]-α-fluoroacetonitrile (4c): ¹H NMR δ 7.15 (d, J=49.15 Hz, 1H), 7.15 (m, 1H), 7.30 (dd, J=7.92, 2.64 Hz, 1H), 7.65 (dd, J=8.91, 4.95 Hz, 1H); ¹⁹F NMR δ -37.55 (m, 1F), -81.11 (d, J=48.72 Hz, 1F); MS *m/e* 226 (M⁺), 199 (M⁺-HCN), 168 (M⁺-CHFCN); HRMS Calcd. for C₉H₄F₂N₂OS: *m/e* 226.0012; Found 226.0022.

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