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The electrolytic method of fluorination in the medium containing the complexes of anhydrous hydrogen fluoride and trialkylamines.

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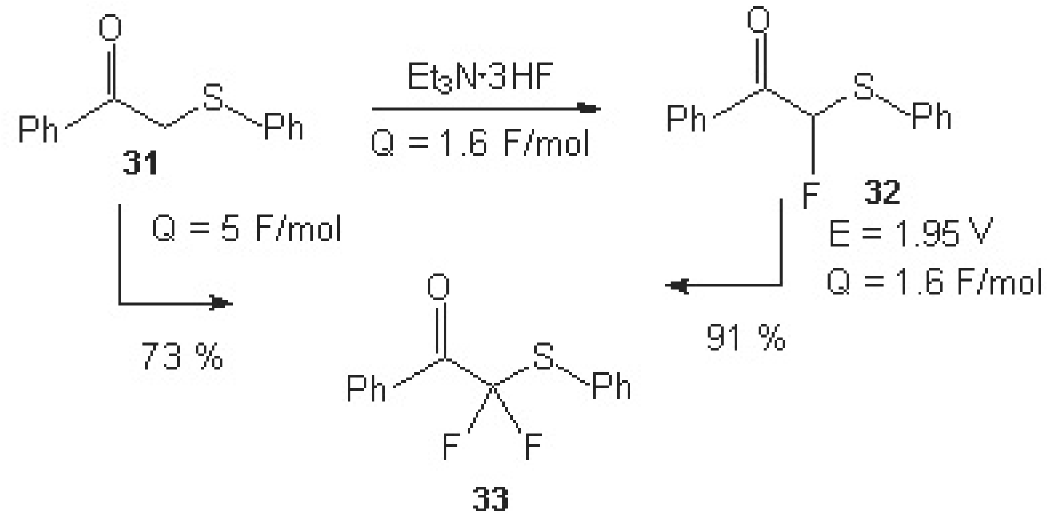
ABSTRACT

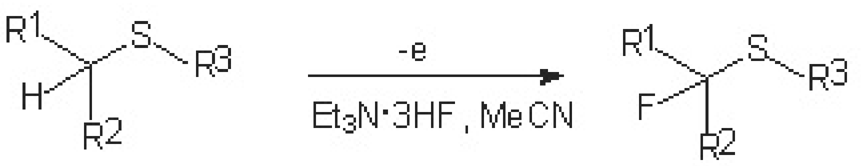
The review generalizes and systematizes the latest date on fluorination of the va
classes of organic compounds by the electrolytic anodic oxidation method in the mediur
electrolyte the complexes of anhydrous hydrogen fluoride and trialkylamines. An analysis
performed of the basic achievements of the method of one atom fluorine introduction. The
having an influence on the process and influence of electron-seeking substitutes arranged by
fragment and by heteroatoms of VIA group are shown. The examples are given of the u
applications of this method for the synthesis of aromatic and heterocyclic compounds as we
compounds having XCHF fragments, where X=S, Se, Te, as the potential intermediate product
synthesis of the preparations possessing a biological activity. The new efficient production met
of monofluorine containing organic compounds and their role in organic synthesis are discusse

Contents

2. Behaviour of organic derivatives of elements of VI group, having CH-fragmen position to heteroatom , in electrolyte solutions, containing anhydrous hydrogen fluo

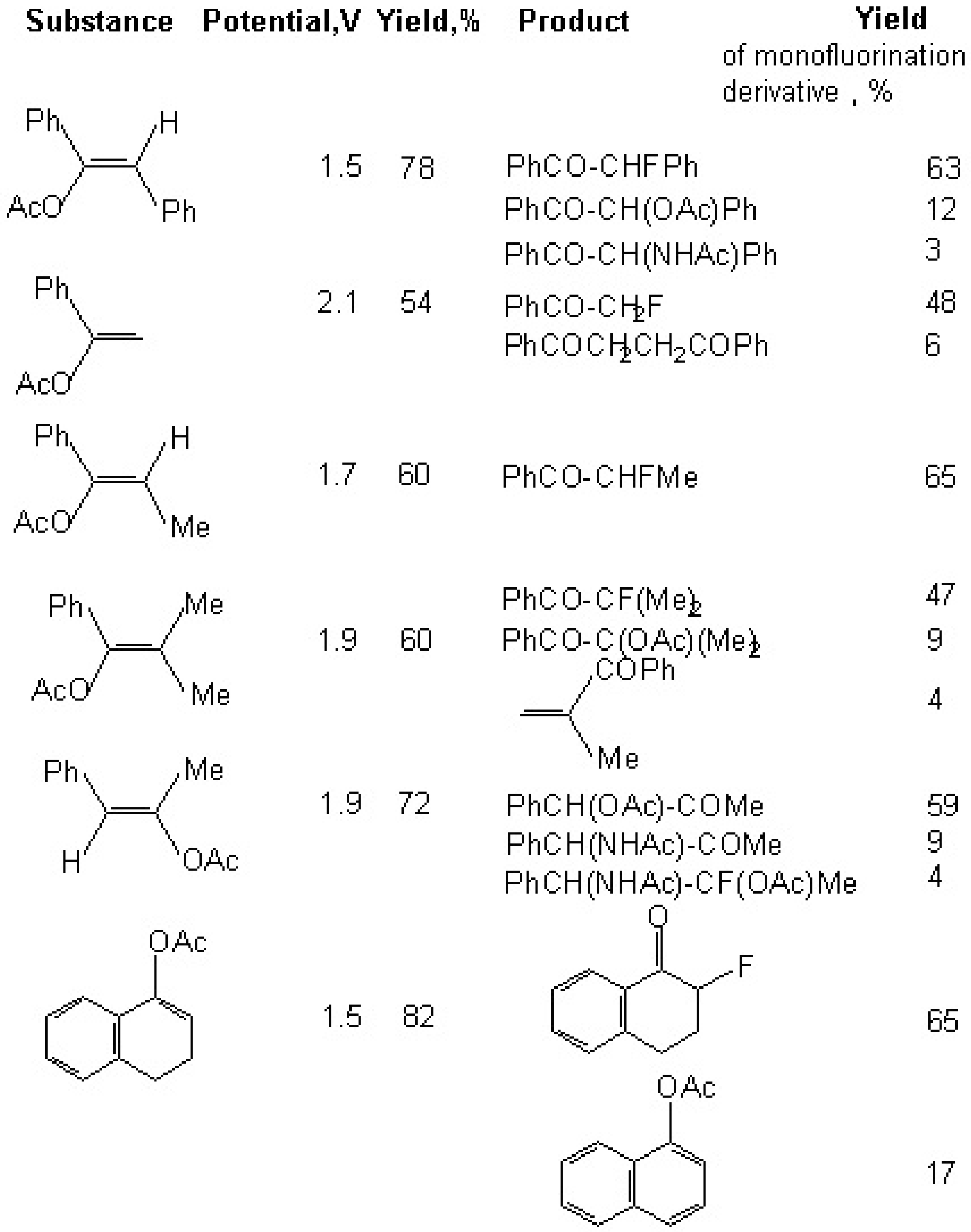
Anodic fluorination of sulfides, containing RCO and Ph groups, in Et_3N* 3HF / MeCN syste 1,1 V results in forming ofα-fluoroderivatives [108]. Thus, monofluoroderivative **32** difluoroderivative **33** are produced out of compound **31**.





Sulfide			E, eV	F/mol^{-1}	Yield, %
R^1	R^2	R^3			
PhCO	H	Ph	1,10	1,6	87
Ph	COOEt	Ph	1,10	6,9	84
COOEt	H	CH_2COOEt	1,46	4,0	50

Table 11. *Organic compounds fluorination (electrolyte Et_3N*3HF, MeCN) [44].*



Substance	Potential,V	Yield,%	Product	Yield of monofluorination derivative , %
	1.5	78	PhCO-CHFPh PhCO-CH(OAc)Ph PhCO-CH(NHAc)Ph	63 12 3
	2.1	54	PhCO-CH$_2$F PhCOCH$_2$CH$_2$COPh	48 6
	1.7	60	PhCO-CHFMe	65
	1.9	60	PhCO-CF(Me)$_2$ PhCO-C(OAc)(Me)$_2$	47 9 4
	1.9	72	PhCH(OAc)-COMe PhCH(NHAc)-COMe PhCH(NHAc)-CF(OAc)Me	59 9 4
	1.5	82		65 17

Other sulfides, containing electron-seeking substituents are also subject to ar monofluorination [64]. The information is given in table 12.

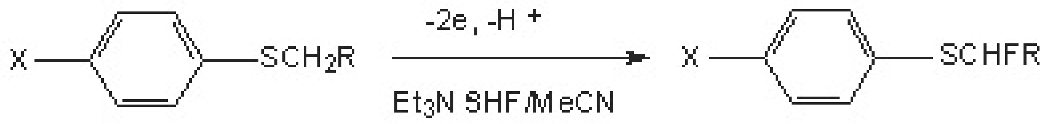
Table 12. *Anodic oxidization of sulfides in the presence of Et3N .3HF/MeCN [64].*

Substrate	Potential of anode, V vs SSCE	F/моль	Product, (yield, %)
C_6H5SCH_2COOEt	1,6	2,5	$C_6H5SCHFCOOEt$ (76)
4-Me$C_6H_4SCH_2COOEt$	1,6	2,1	4-Me$C_6H_4SCHFCOOEt$ (78)
$PhCH_2SCH_2COOEt$	2,1	5,0	$PhCH_2SCHFCOOEt$ (44)
n-$C_7H_{15}SCH_2COOEt$	2,1-2,3	16,1	n-$C_7H_{15}SCHFCOOEt$ (70)
$PhSCH_2CN$	1,7	5,0	PhSCHFCN (75)
$PhSCH(COOEt)_2$	2,0	15,4	$PhSCF(COOEt)_2$ (77)
$PhSCH_2COMe$	1,6-1,8	7,6	PhSCHFCOMe (80)
$PhSCH(COMe)_2$	1,7	3,0	PhSCHFCOMe(55)

Fuchigami with colleagues has done his bit in understanding of anodic fluorination pro passing ways [100,109].

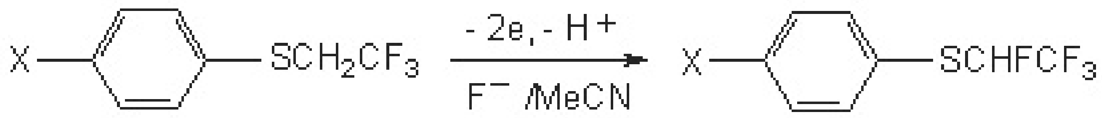
The presence of phenyl or the electron-seeking substituent in α-position to sulfur ator necessary [48,64,108,110].

Table 13. *Anodic fluorination of arylalkylsulfides [110].*



X	R	Ep^{ox}	Potential of anode, V	Q, F/mol	Yield, %
MeO	CF_3	1,49	1,7	6,0	56
H	CHF_2	1,69	2,3	4,0	53
H	CH_2F	1,58	2,1	2,7	60
H	CF_2Cl	1,80	2,0	5,0	43
H	H	1,51	1,6	4,0	27
H	CH_3	1,48	1,8	4,0	18

The system in which anodic fluorination is conducted is really important. Thus, by examp anodic monofluorination of aryl-2,2,2-trifluoroethylsulfide the increasing of fluorination product at transfer from Py* n(HF) system to Et_3N* 3HF [110] system is registered.



X	electrolyte	Potential of anode, V vs SSCE	F/mol	Yield, %
H	Py*nHF	+2.0	4,1	0
H	Bu_4NF*3H_2O	+2.0	1,9	0
H	Et_3N*3HF	+1.9	3,2	62
Cl	Et_3N*3HF	+2.0	7,2	65
Me	Et_3N*3HF	+2.1	8,2	51

It is interesting that anodic fluorination of benzylfluoroalkylsulfides in Et_3N* 3HF/MeCN sys results in forming of two regio-isomers [110].

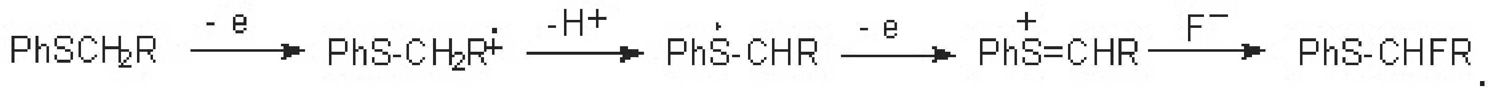
$$PhCH_2SCH_2R_F \xrightarrow[Et_3N \cdot 3HF\ /\ MeCN]{-2e,\ -H^+} PhCH_2SCHFR_F + PhCHFSCH_2R_F$$

R_f	Potential of anode, V vs SSCE	F/mol	Products yield, %	
			$PhCH_2SCHF>R_f$	$PhCHFSCH_2R_f$
CF_3	+2.5-2.8	4.0	22	32
CF_3	+2.3	11.5	26	45
CF_3	+2.3	17.0	51	30
CH_2F	+2.5-2.7	3.0	13	34

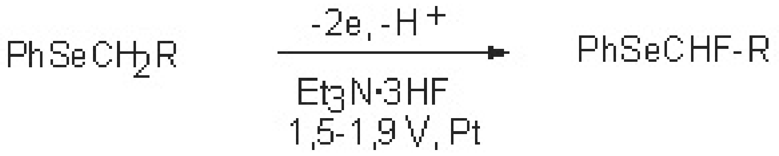
Selective anodic fluorination of ethylphenylsulfanylacetate can be conduct in the flawelectrolyzer at constant potential as well as in galvanostatic mode using anodes from OPTA, carbon in electrolyte Et_3N* 3HF and current density 15 mA/cm^2 (yield of monofluorination pro is 61, 51 and 49 % accordingly) [98]. Anodic fluorination of sulfides, having electron-see substituents iα-position to sulfur atom (such groups as cyan, ethers, acyl and phospha enables to introduce selectively fluorine atom into the same position [97].

The highest yield of fluorination products is achieved in case of fluorination of $PhSCH_2CO$ (88 %) [97], 4-$MeC_6H_4SCH_2COOEt$ (78 %) [111] and $PhSCH_2COPh$ (87 %) [108]. Mentioned can be successfully used for fluorination cα-phenylthiosubstituted cyclic carbonyl compound well as for obtaining (α,-difluoroderivatives of ethα-phenylthioacetate

The monofluorination mechanism of sulfur-containing compounds was not adequately explo [6,8,9,64]. Many authors consider, that the process includes one-electrone oxidizatio substrates with forming of cation-radical. Subsequent steps of the process can pass in two: e by elimination of proton with generation of active radical, which oxidizes to cation under the a of current with generation of sulfinic cation, which later reacts with fluoride-ion with formir reaction product or direct attack of carb-cation center by fluoride-ion occures



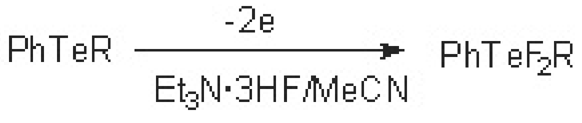
Selenium-containing compounds also react the same (table 14) [15,64,100,109,112-116].



R = CN (71 %), COOEt (70 %),
$COCH_3$ (37 %), $CONH_2$ (60 %

The best results for compounds $XC_6H_4SeCH_2R$ (X = H, Cl, MeO) are achieved if electron-see groups act as **R** (R = CN, COOEt, CONH2) [116].

At the same time anodic fluorination of tellurium derivatives in Et_3N*3HF (Pt electrodes, cu density 12.5 mA/cm^2 , anodic potential 1,5 V) in conditions of galvanostatic electrolysis do result the substitution of hydrogen, which iα-position to tellurium, but the fluorination of tellu atom occures, accompanied by its oxidization up to quadrivalent state and forming difluoroderivative [117]. At that the nature of solvent slightly influences the yield of final fluorin product.



	Solvent	Yield,%
R = CH_2CF_3	MeCN	86
	MeOH	75
	AcOH	70
R = Me	MeCN	81
	AcOH	78
R = Ph	MeCN	77
	AcOH	75

Interesting example of electrolytic fluorination is described in works [69,118], when at ca atom there are two phenyl groups and two SPh groups. In this case the substitution of SPh gr for fluorine atoms occurs .

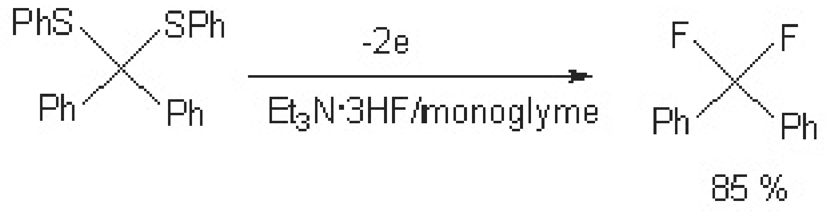
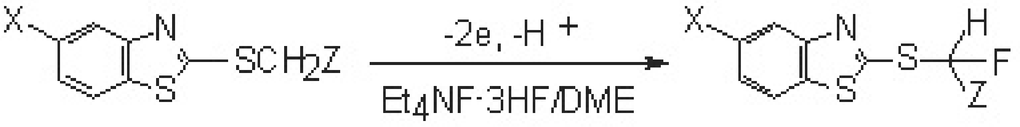


Table 14. *Anodic fluorination of phenylselenides [64].*

Substrate	Potential of anode,V	F/mol	Yield, %
$PhSeCH_2CN$	1,6-1,8	6.6	71
$PhSeCH_2COOEt$	1.5	5.8	70
$ClC_6H_4SeCH_2COOEt$	1.8-1.9	6.5	81
$PhSeCH_2COCH_3$	1.6-1.7	3.5	37
$PhSeCH_2CONH_2$	1.5-1.6	3.5	60
$PhSeCH_2Ph$	1.3-1.5	20	traces
$PhSeCH(CH_3)COOEt$	1.6	3.1	3
$PhSeCH(CH_3)CN$	1.7-2.0	3.3	16
$PhSeCH(COOEt)_2$	1.8	2.7	55
$PhSeCHClCOOEt$	2.3	3.0	65

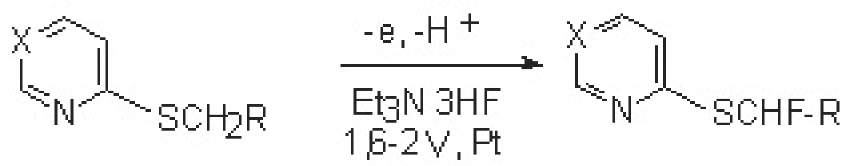
Anodic desulfurization of ketone dithioacetals in the presence of Et_3N*3HF system prod hem-difluorothioethers and monofluorothioethers [69]. Thereby it is necessary to use behavior of sulfur-containing compounds carefully. At the same time this is an intere substitution example of sulfur-containing group for fluorine atom.

Sulfur-containing substitutes influence on anodic fluorination process is typical not onl aromatic compounds, but also for heterocyclic ones. Thus, electrolytic fluorination o benzothiazolyl- and 5-chloro-2-benzothiazolyl- sulfides using Et_4NF*3HF system dimethoxyethane (DME) produces appropriatα-monofluorinated sulfides [119].



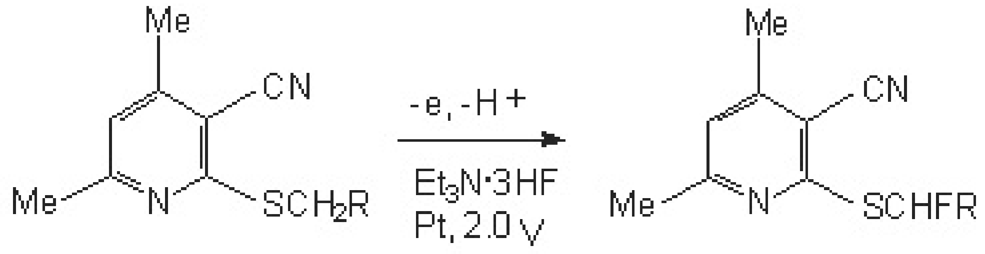
X	H	H	H	Cl	Cl	Cl
Z	CN	CO_2Me	COMe	CN	CO_2Me	COMe
Yield, %	48	62	46	51	82	58

Laurent [108] and Fuchigami with their colleagues [64,86,109,120] discovered the forma ofα-monofluoroderivative sulfides during anodic selective fluorination of nitrogen-conta heterocyclic sulfides, which have electron-seeking substituent in methene group. Thus, conduction of impulse electrolysis of pyridine and pyrimidine derivatives in Et_3N*3HF/M medium with Pt electrodes at 25 ^{o}C results in formation of monofluoroderivatives [114,120].



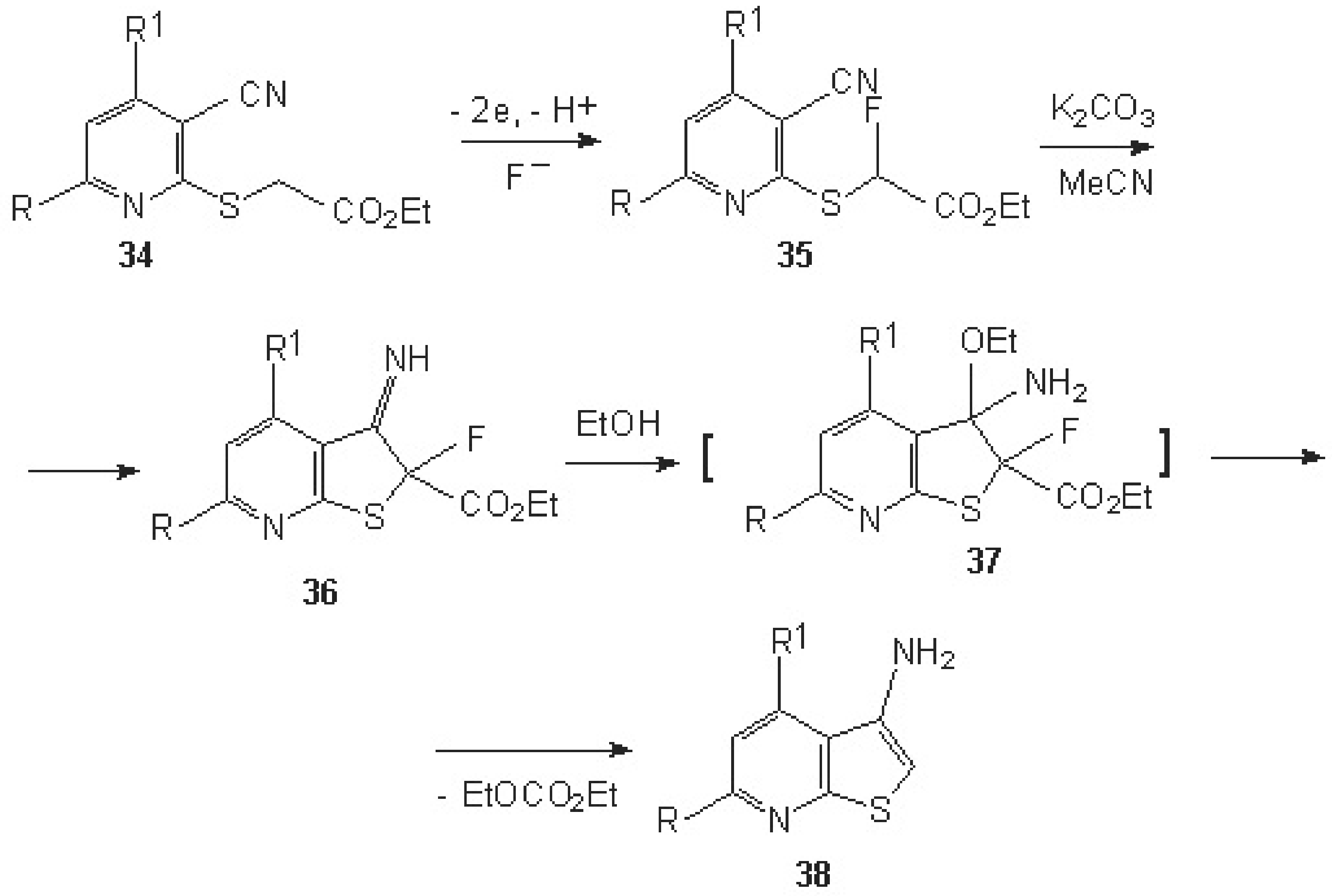
X	R	Potential of anode, V vs SSCE	F/mol	Yield, %
CH	CN	1.6	5	76
CH	COOEt	1.6	5	76
CH	H	1,6	5	traces
CH	CH_2CN	1.6	6	traces
CH	$PO(OEt)_2$	1.6	6	traces
CH	SMe	1.6	5	traces
N	COOEt	2.2	11	55

Pyridine derivatives, having iα-position SCH_2R group, are also subject to anodic fluorination formation of monofluoroderivatives [114].



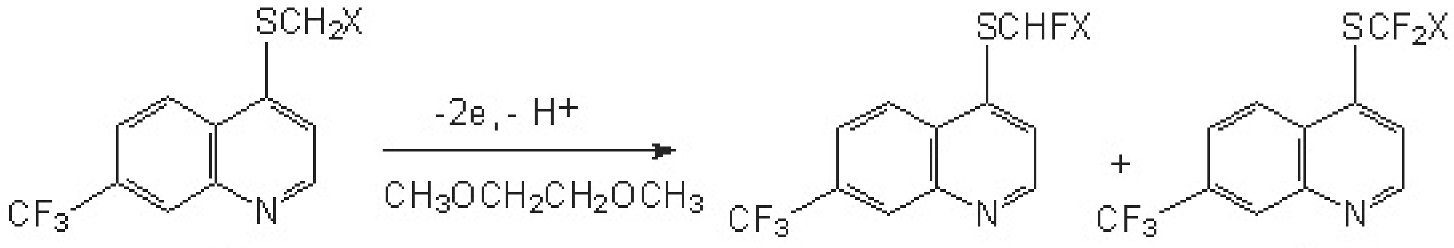
R = CN (55 %), COOEt (45 %)

The presence iα-position CN group (2-**pyridylsulfide 34**) allows to obtain (in the presenc base) bicyclic heterocycle 2-fluorothieno[2,3-b] pyridine **38**. The reaction must pass as follow firsα-**fluoromethylpyridylsulfide 35** forms due to anodic fluorination of 2-**pyridylsulfide** Thienopyridinimine **36** is formed in the base medium. This compound reacts with ethyl alcohol produces intermediate compound **37**, which aromatization with elimination of diethylcarbo results in formation of compound **38**.



We'll note, that fluorination of these heterocyclic compounds by such fluorine reagent triphthalate N-**fluoropyridinium** and N-fluoro-3,5-dichloro**pyridinium** proved to be ineffe [121].

Electrochemical anodic fluorination of 4(7-trifluoromethyl)quinolylsulfides **39** was realize $Et_4NF*nHF$ (n=3,4) and Et_3N*3HF systems in dimethoxyethane. Correspondi monofluoroderivatives of these sulfides **40** are obtained with good yield (table. 15) [122]. Fo case X = COMe the formation of compound **41** take place too (yield 7 %).



39 **40** **41**

For 2-quinolylsulfides **42** the formation of three compounds **43-45** take place (table 16).

Table 15. *Anodic fluorination of 4(7-trifluoromethyl)quinolylsulfides (dimethoxyethane) [122*

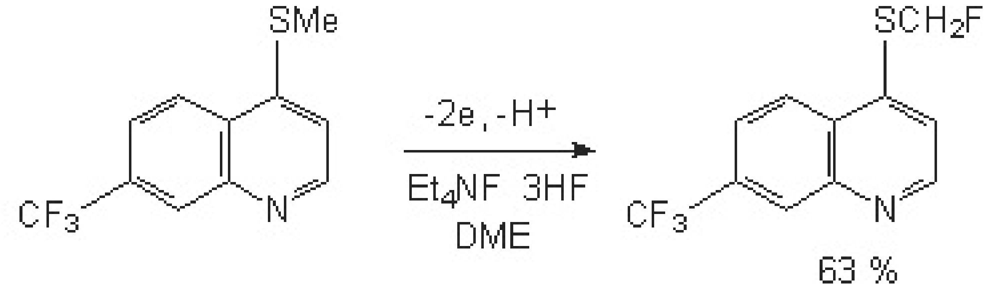
X	Eoxp (V vs SSCE) Pt-electrode 0,1M Bu_4NBF_4	Electrolyte	F/mol	Yield of 40,%
COMe	1,92	$Et_4NF*3HF$	4	96
		$Et_4NF*4HF$	2,5	62
COOEt	2,04	$Et_4NF*3HF$	4,5	83
		Et_3N*3HF	6	67
CN	2,14	$Et_4NF*4HF$	4	86
		$Et_4NF*3HF$	4	87
		Et_3N*3HF	6	77

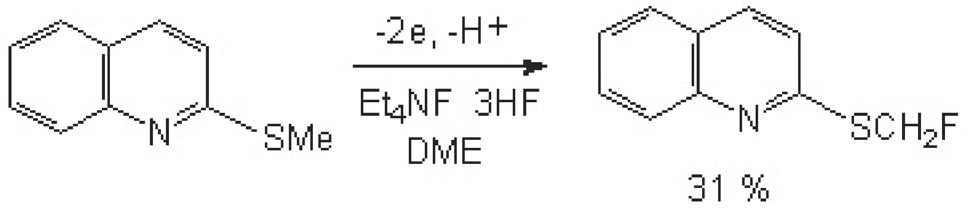
Table 16. *Anodic fluorination of 2-quinolylsulfides [122]*

X	Eoxp (V vs SSCE) Pt-electrode 0,1M Bu_4NBF_4	Electrolyte	F/mol	Product's yield, %		
				43	44	45
COMe	1,7	$Et_4NF*4HF$	4	54	8	5
		$Et_4NF*3HF$	6	50	8	5
		Et_3N*3HF	7	55	8	5
		Et_3N*3HF	6	64	-	-
		Et_3N*3HF	4	28	-	-
COOEt	1,76	$Et_4NF*4HF$	4	62		5
		$Et_4NF*3HF$	4	64		5
		Et_3N*3HF	6	59		7
CN	1,91	$Et_4NF*4HF$	8	61		12
		$Et_4NF*3HF$	8	52		12
		Et_3N*3HF	8	62		12

The solvent's influence on this reaction has been studied by Fuchigami [123]. It turned out, dimethoxyethane is the best solvent, acetonitrile is less effective though it’s mixture dimethoxyethane gives quite good results.

If in heterocyclic ring there is SMe groups the poor yield substitution of hydrogen from m group by fluorine take place.



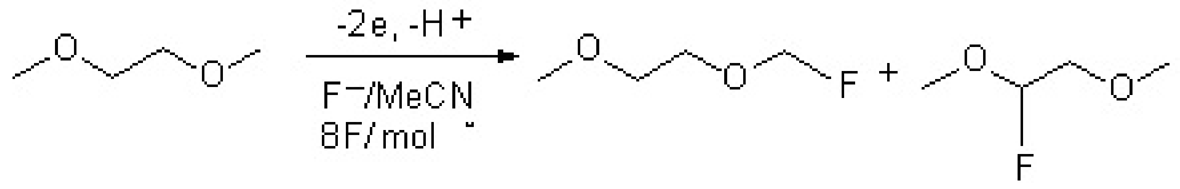


It should be noted, that such acknowledged fluorinating agents as N-fluoropyridinic salts [showed absolute inertness at present objects. This allows to consider electrochemical fluorin as alternative method for known fluorination processes and its further development application to other organic substrates can be expected.

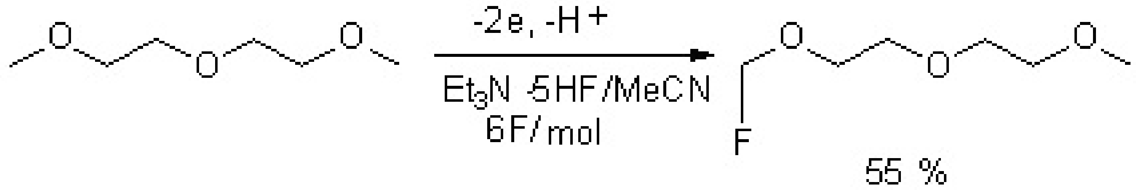
Thus, the new approach to synthesis of hard-to-reach derivatives of sulfur and selenium wi CHF и SCF$_2$, Se-CHF fragments is developed, some of which are high biologically active [121 spite of seemed vagueness regarding commercial application the fluorination by electr method continues to attract active interest of researchers. The opportunity of moving fluorination centre from aromatic cycle onto functional groups, containing, for example a methene group, creates real perspectives for development of convenient synthesis metho hard-to-reach monofluoroderivatives. Polyfluorinated hydrocarbon synthesis using this me seems to be workable, that by all means is important.

α-Fluorocontaining derivatives of aromatic compounds with C=O and S-R-groups are increas biologically active. Their synthesis is carried out by fluorination using xenon difluoride [125-1 DAST [124,128], triphthalate N-**fluoropyridinium** , particularly N-fluoro-2 trimethyl**pyridinium** triphthalate and N-fluoro-3,5-dichloro**pyridinium** triphthalate [However these methods give lower yields and poor results comparing to anodic fluorin method.

Dimethoxyethane and dimethyl ether of diglycol at anodic fluorination in electrolytes Et$_3$N and Et$_4$NF*4HF give either mixtures of isomeric fluoroethers or monofluoroderivative with flu atom located in the beginning of carbon chain [61].



electrolyte	yield, %	
$Et_3N\cdot 3HF$	31	24
$Et_3N\cdot 5HF$	62	14
$Et_4NF\cdot 3HF$	49	11
$Et_4NF\cdot 4HF$	59	19



As a whole, electrochemical fluorination method as way to low-fluorinated derivatives conti to attract the attention of researchers, in spite of today's vagueness regarding comme application.

Conclusion

The presented material allows to make a conclusion that electrochemical monofluorinatic electrolytes - the complexes of anhydrous hydrogen fluoride and trialkylamines - is an impor method of obtaining of monofluorinated organic compounds, which opportunities steadily incr as far as present method improves. At this different classes of compounds are entered reaction, that resulted in noticeable success of aromatic and heterocyclic monofluorin derivatives synthesis. The materials obtained have found practical application. Becaus importance of these organic compounds classes the improvement tasks of their obta technology are urgent and stimulated by requirements of techniques. Obviously, the fu research in this field will go on both in extension of started before aspects of electroche process itself and in transfer to fundamentally new decisions and approaches. Here it is impor not only to formulate common rules of organic compounds behavior in electrochemical fluorin process but also try to reveal the specific particularities properties of the process, cause fluorine atoms. Such substrates, as can be seen from stated above, are noticeable persistent in anodic oxidization conditions and give noticeable higher outputs, that no d simplifiers the stage of target product extraction. Thorough researches for development o knowledge of electrochemical fluorination mechanism should mean a lot.

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