

## Preparative Electrochemical Reduction of Fluorinated Benzonitriles

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**Abstract**—Electrochemical reduction of fluorinated benzonitriles in aprotic media is accompanied by concurrent processes of dimerization and fragmentation of the corresponding anion-radicals. With 2-fluoro- and 3,4,5-trifluorobenzonitriles the defluorinated products can be obtained at direct electrochemical reduction; for the other compounds studied have been found mediators providing a possibility to obtain in high yield defluorinated products at preparative electrochemical reduction. A fundamental possibility was demonstrated of providing products by nucleophilic fluorine substitution by  $S_{\text{BN}}$  mechanism at electrochemical reduction of fluorinated benzonitriles in aprotic solvents in the presence of mediators.

Recently the electronic structure and reactivity of anion-radicals from haloaromatic compounds are extensively studied (see, e.g. [1–4]). The interest to anion-radicals of this type is due to a great extent to the fact that their fragmentation to aryl radicals and halide anions constitutes the elementary stage of nucleophilic substitution of the  $S_{\text{RN}}1$  type and of reductive halogenation.

We investigated previously by means of cyclic voltammetry the electrochemical reduction of a large series of fluorinated benzonitriles in DMF [5–7]. It was established that all fluorinated benzonitriles studied underwent in DMF one-electron electrochemical reduction providing anion-radicals of different stability. For instance, anion-radicals of 3-fluoro- and 3,5-difluorobenzonitriles are relatively stable in DMF: On the cyclic voltammetric curves of these nitriles in the system DMF-tetraethylammonium perchlorate (TEAP) reversible one-electron reduction peaks are present, and at reduction of the nitriles in a cell placed in the probe of an ESR-spectrometer well resolved ESR spectra of the corresponding anion-radicals are observed [5].

Anion-radicals of benzonitriles with a fluorine atom located in the *para*-position to the cyano group suffer as concurrent processes elimination of the *para*-located fluorine and dimerization at this position [6] (Scheme 1).

The anion-radicals with hydrogen atom in the *para*-position to cyano group undergo fragmentation at the *ortho*-position, and concurrent dimerization at the *para*-position is here reversible (Scheme 2). Basing on these data we investigated in this work the possibility of preparative electrochemical reduction of fluorinated benzonitriles.

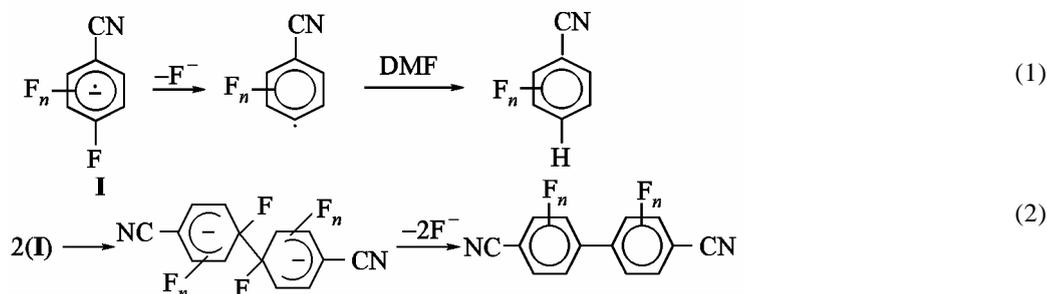
It follows from the above stated that the electrochemical reduction of fluorinated benzonitriles can provide defluorination product only if the parallel dimerization process of the corresponding anion-radicals is reversible, as with 2-fluorobenzonitrile, and if the latter process is irreversible as with compounds having a *para*-located fluorine, it should be considerably slower than fragmentation. Actually, the preparative electrolysis of 2-fluorobenzonitrile furnishes as main product the benzonitrile in 80% yield.\*

From 3,4,5-trifluorobenzonitrile we also obtained the product of anion-radicals fragmentation, 3,5-difluorobenzonitrile (59%).

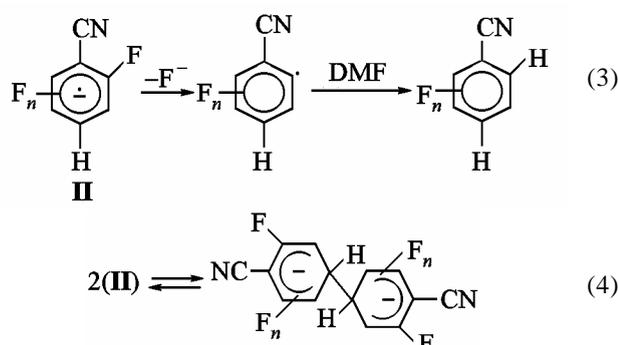
The fraction of dimerization products obtained in the mentioned processes depends on the rates ratio of concurrent transformations of anion-radicals that in its turn is affected by the concentration of the initial com-

\* Here and hereinafter the yield of products is given with respect to the initial compound.

Scheme 1.

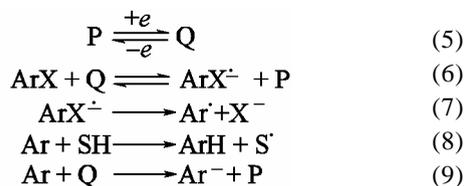


Scheme 2.



pond. Therefore with certain fluorinated benzonitriles it is possible to choose concentration where the fragmentation of anion-radicals is the prevailing process. For instance, at concentration of 4-fluorobenzonitrile of  $10^{-4}$  M the main product of electrochemical reduction is benzonitrile [8]. However not always the decrease in concentration completely prevents dimerization of anion-radicals.

It was presumable that the ratio of fragmentation and dimerization products may be changed also at the use of mediators. The principal stages of the mechanism of indirect reductive electrolysis may be represented by reactions sequence (5)–(9) [9].



Here P and Q are respectively neutral and anion-radical forms of mediator, and SH is solvent.

In this case the electron transfer to substrate occurs not at the electrode surface but in solution of the mediator which is present in a concentration considerably

lower than that of the substrate. Therefore the concentration of the arising anion-radicals would be significantly lower than that required for notable competition of dimerization with fragmentation.

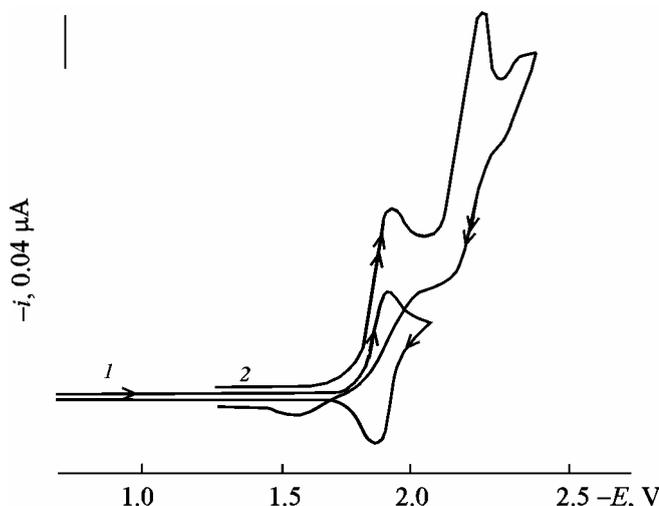
In selection of mediator the following criteria should be used [10, 11]:

- (1) the reduction potential of the mediator should be less negative than that of the compound under study;
- (2) stability of the anion-radical form of mediator Q;
- (3) sufficiently high rate of electron transfer from the Q form to substrate.

Criterion (1) means that electron transfer from the anion-radical of mediator to the substrate is thermodynamically unfeasible, but irreversibility of reaction (7) ensures the occurrence of the process.

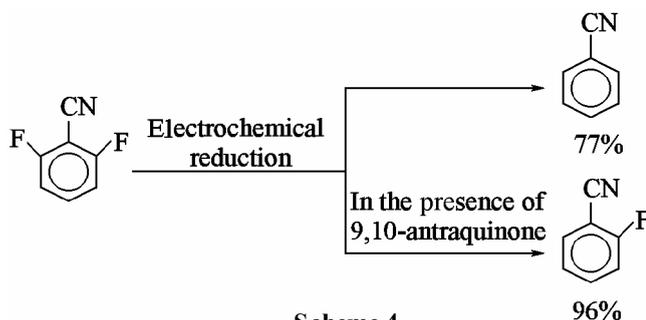
We proved the possibility to apply mediator for prevailing occurrence of fragmentation by an example of 4-fluorobenzonitrile. The direct electrochemical reduction of the latter at  $c > 10^{-3}$  M is accompanied by dimerization [8]. The efficiency of mediator was estimated by cyclic voltammetry. On the figure is shown a cyclic voltammogram of a potential mediator, anthracene. The curve contains a reversible one-electron peak of anthracene reduction into anion-radical ( $I_a/I_c = 1$ ). The addition of 4-fluorobenzonitrile to the system results in increase of the cathode wave and in decrease of the  $I_a/I_c$  ratio as a consequence of electron transfer from the anion-radical of anthracene to the 4-fluorobenzene molecule with anthracene recovery along reaction (6). This result evidences that anthracene can be applied as mediator. Actually, the main product of 4-fluorobenzene electrolysis in the presence of anthracene was benzonitrile (see Table 1) that corresponded to the fragmentation of the anion-radical as the prevailing process.

The electrochemical reduction of 2,4-difluorobenzonitrile and 2,6-difluorobenzonitrile furnished

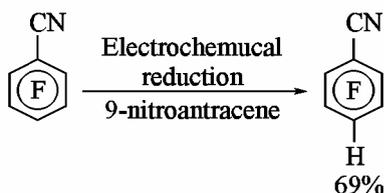


Cyclic voltammogram of  $10^{-1}$  M anthracene solution in  $\text{CH}_3\text{CN}$ , supporting electrolyte 0.1 M TEAP,  $v$   $50 \text{ mV sec}^{-1}$  (1) with no 4-fluorobenzonitrile, (2) in the presence of 4-fluorobenzonitrile.

Scheme 3.



Scheme 4.



benzonitrile. In both cases no product of a single fluorine elimination, 2-fluorobenzonitrile, was found. Apparently the latter product underwent reduction under experimental conditions for the reduction potentials of 2,4-difluoro- and 2-fluoronitriles are virtually equal (in DMF  $E_0$   $-2.04 \text{ V}$ ), and those of 2,6-difluoro and 2-fluorobenzonitriles are sufficiently close (in DMF  $-1.90 \text{ V}$  and  $-2.04 \text{ V}$  respectively). In the first case in the products mixture were found also unidentified substances that apparently originate from further electrochemical reduction of dimerization products (Scheme 3).

It was presumable that application of mediator would decrease the probability of dimerization. Actually, the electrochemical reduction in the presence of 4-cyanopyridine as mediator resulted in reaction of 2,4-difluorobenzonitrile in a product mixture containing 70% of benzonitrile and no products with a double molecular weight. Besides the properly selected mediator and the use of redox-catalysis permits prevention of reduction of the primary defluorination product since the equilibrium constant of the homogeneous electron transfer ( $K$ ) depends essentially on the difference between the standard reduction potentials of the mediator and substrate,  $\hat{E} = \exp(-F\Delta E_0/RT)$  [12], and even small (0.1–0.2 V) changes in the  $\Delta E_0$  value significantly affect the rate of electron transfer. In keeping with above at electrochemical reduction of 2,6-difluorobenzene with 9,10-antraquinone as mediator at reduction potential corresponding to reduction of the anthraquinone anion-radical to dianion a single fluorine atom was successfully eliminated, and 2-fluorobenzonitrile was obtained (Scheme 3).

As showed the experiment with pentafluorobenzonitrile, the accumulation of fluorine atoms in the benzene ring led to complication of the process by polymerization and tarring on the electrodes. As a result it is impossible to carry out the reaction in a preparative mode. It was attempted to overcome these difficulties by application of a mediator. To the requirements given above for mediators correspond nitrobenzene, 4-nitrotoluene, 9,10-antraquinone, 9-nitroanthracene. Although at electrochemical reduction of pentafluorobenzonitrile in the presence of first two of the mentioned mediators the product of fluorine elimination from the *para*-position with respect to cyano group was obtained, but its yield was very small (see Table 1). This fact apparently shows that the yield of reaction products depends not only on the difference in  $E_0$  of the mediator and the substrate but also on the structure of the anion-radicals used. On the contrary the electrochemical reduction of the pentafluorobenzene in the presence of 9-nitroanthracene resulted in successful selective elimination of fluorine atom only from the *para*-position at complete conversion of the initial compound (Scheme 4).

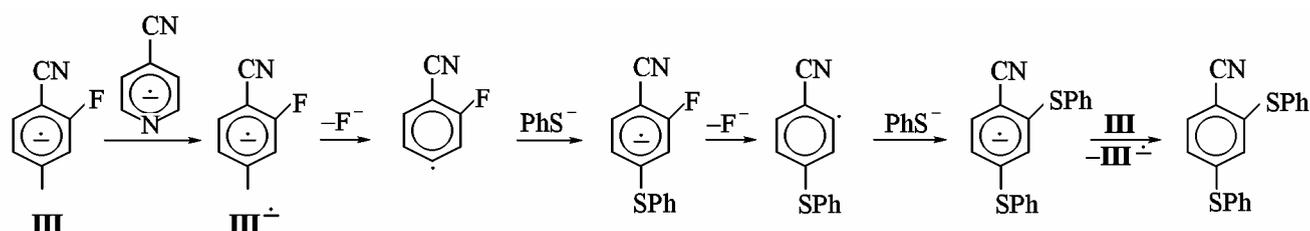
Thus it was established that in preparative mode of electrolysis 2-fluoro and 3,4,5-trifluorobenzonitrile provide the desired product at direct electrochemical reduction. For the other studied benzonitriles were selected mediators that provide a possibility to direct the

**Table 1.** Experimental conditions and products of preparative electrochemical reduction of fluorinated benzonitriles  $C_6F_nH_{5-n}CN$ 

Substituents	Amount of compound, mg (concentration, $\dot{I}$ )	$E_{red}$ , V	Electrolysis time, h	Mediator [amount, mg, (concentration, M)]	Composition of reaction mixture (according to GLC data)			Solvent																							
					initial compound, %	defluorination product, %	dimerization product, %																								
2-F	60 ( $5 \times 10^{-2}$ )	-2.10	3	Anthracene [ $2 (1 \times 10^{-3})$ ]	52	80	Polymeric products	DMF																							
4-F	43.5 ( $7.2 \times 10^{-2}$ )	-2.20	3.25			MeCN		15	43	MeCN																					
	61.2 ( $1 \times 10^{-1}$ )	-1.85	3.5						DMF		15	21.5	MeCN																		
2,4-F <sub>2</sub>	74 ( $5.3 \times 10^{-2}$ )	-2.20	3.25									70		23	70	DMF															
	70 ( $5 \times 10^{-2}$ )	-1.80	3												4-Cyanopyridine [ $5.2 (5 \times 10^{-3})$ ]		37	77	MeCN												
2,6-F <sub>2</sub>	71 ( $7 \times 10^{-2}$ )	-1.86	4.25															95.7		17.7	59.3	DMF									
	20.7 ( $2 \times 10^{-2}$ )	-1.45	8.25																		9,10-Anthraquinone [ $4 (2 \times 10^{-3})$ ]		18.5	1.1	MeCN						
3,4,5-F <sub>3</sub>	100 ( $9.1 \times 10^{-2}$ )	-1.85	3																					17.7		18.5	1.5	MeCN			
	67.5 ( $5 \times 10^{-2}$ )	-1.27	3.5																								4-Nitrotoluene [ $1.4 (10^{-3})$ ]		50	0	MeCN
	67.5 ( $5 \times 10^{-2}$ )	-1.23	3.5																											Nitrobenzene [ $1.2 (10^{-3})$ ]	
	67.5 ( $5 \times 10^{-2}$ )	-0.95	3	9,10-Anthraquinone [ $2 (10^{-3})$ ]	67.5		16																								
	67.5 ( $5 \times 10^{-2}$ )	-0.94	2.25			4,4'-Dinitrodiphenyl [ $2.5 (10^{-3})$ ]	34.3	0		MeCN																					
		-1.05	2.25					9-Nitroanthracene [ $2 (10^{-3})$ ]	69				MeCN																		
		-1.10	4.5													MeCN															
	61.5 ( $5 \times 10^{-2}$ )	-1.10	3												MeCN																

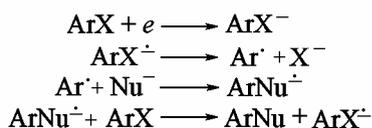
**Table 2.** Experimental conditions and products of preparative electrochemical reduction of fluorinated benzonitriles  $C_6F_nH_{5-n}CN$  in the presence of 0.1 M tetrabutylammonium thiophenolate

Substituents	Amount of compound, mg (concentration, $\dot{I}$ )	$E_{red}$ , V	Electrolysis time, h	Mediator [amount, mg, (concentration, M)]	Composition of reaction mixture (according to GLC data)			Solvent		
					dimerization product, %	defluorination product, %	product of nucleophilic substitution, %			
2-F	60 ( $5 \times 10^{-2}$ )	-2.10	3	4-Cyanopyridine [ $5.2 (5 \cdot 10^{-3})$ ]	+	80	20	DMF		
2,4-F <sub>2</sub>	70 ( $5 \times 10^{-2}$ )	-2.10	3			MeCN	+	+	(PhS) <sub>2</sub> C <sub>6</sub> H <sub>3</sub> CN	
	70 ( $5 \times 10^{-2}$ )	-1.80	3					MeCN		15

**Scheme 5.**

transformation of anion-radicals on the fragmentation route and thus to obtain in sufficiently high yield  $\delta$ -fluorination products.

As was already mentioned, the fragmentation of anion radicals of the haloaromatic compounds is the key stage of nucleophilic substitution reactions of  $S_{RN}1$  type [13].



Many examples are known of the use of compounds  $\text{ArHlg}$  ( $\text{Hlg} = \text{I}, \text{Cl}, \text{Br}$ ) in this type reactions, but with substituted fluoroarenes exist only few examples [14]. With fluorinated benzonitriles the reactions of nucleophilic substitutions proceeding along  $S_{RN}1$  mechanism may be complicated due to irreversible dimerization of anion-radicals, but we hoped that this can be avoided by using mediators.

Dimerization of anion-radicals of 2-fluorobenzonitrile is reversible and therefore should not bring about the mentioned complications. Therefore we tried to carry out the nucleophilic fluorine substitution in 2-fluorobenzonitrile at direct electrochemical reduction. The cyclic voltammogram showed that in the electrochemical reduction of this compound in the presence of tetrabutylammonium thiophenolate occurred fluorine substitution with a thiophenyl group as evidenced by appearance of oxidation wave of 2-phenylthiobenzonitrile [7]. The preparative electrolysis of 2-fluorobenzonitrile in the presence of  $\text{Bu}_4\text{NSPh}$  as supporting electrolyte furnished benzonitrile and 2-phenylthiobenzonitrile in 80 and 20% yield respectively. The low yield of the product of nucleophilic substitution is caused by competition in DMF between the trapping of the cyanophenyl radical with the nucleophile and hydrogen rupture from the solvent molecule by this radical [15].

The direct preparative electrochemical reduction of 2,4-difluorobenzonitrile did not give any product of nucleophilic substitution at all. This is apparently due to the high rate of fragmentation of the anion-radical from 2,4-difluorobenzonitrile ( $k = 10^2 \text{ s}^{-1}$  [6]). As a result the anion-radical arising on the electrode has not enough time to pass into the volume of the solution, and the cyanophenyl radical formed close to the surface of the electrode further undergoes reduction into anion. An

addition complication consists in irreversible dimerization as show compounds with a double molecular weight among the products. It was presumable that the encountered complications might be avoided by using a mediator since in this case the reduction of nitrile would occur in solution and not on the electrode. Then the probability of reduction of the cyanophenyl radical produced by fragmentation of the primary anion-radical would be a lot smaller, and its main reaction would be that with thiophenolate anion. The reaction of 2,4-difluorobenzonitrile with the thiophenolate anion initiated electrochemically was carried out with 4-cyanopyridine as mediator in acetonitrile solution at the reduction potential of the cyanopyridine. As the main product was obtained in 70% yield the compound originating from the double substitution, 2,4-diphenylthiobenzonitrile. This result can be rationalized as follows: The intermediately arising anion-radical of 2-fluoro-4-phenylthiobenzonitrile is unstable and undergoes fragmentation with elimination of fluorine in the *ortho*-position with respect to cyano group transforming into 4-phenylthiocyanophenyl radical that further reacts with the thiophenolate anion giving the product of double substitution (Scheme 5).

Thus the results obtained demonstrate the promising prospects of applying mediators in reactions of reductive defluorination and aromatic nucleophilic substitution along  $S_{RN}1$  mechanism for fluorinated aromatic compounds.

## EXPERIMENTAL

Cyclic voltammograms were registered on a modified polarograph LP-7e equipped with three-electrode cell. As the main electrode served a platinum disk of  $0.07 \text{ cm}^2$  surface area, the supplementary electrode was of a platinum spiral, and as reference electrode was used a saturated calomel electrode. Electrochemical measurements were performed with solutions of the initial compounds in DMF or acetonitrile of concentrations  $(1-5) \times 10^{-3} \text{ M}$ , supporting electrolyte 0.1 M tetraethylammonium perchlorate. To remove oxygen the cell and solutions were flushed with argon. Anhydrous DMF was prepared by double vacuum distillation over phosphorus pentoxide collecting the fraction of bp  $34^\circ\text{C}$  (1 mm Hg). The acetonitrile was purified by distillation over potassium permanganate followed by double distillation over phosphorus pentoxide. The solvents were stored over molecular sieves.

The preparative electrolysis was carried out with the use of a potentiostat P-5848 in a potentiostatic mode in a three-electrode cell. As cathode served a

platinum plate of 1 cm<sup>2</sup> surface area, as anode a platinum spiral.

As a reference electrode was used a saturated aqueous calomel electrode. After treatment the reaction mixture containing the products of electrochemical synthesis were analyzed by GLC on a chromatograph LKhM-72 under the following conditions: stationary phase 15% of SKTFB-803 on Chromosorb W, oven temperature programmed in the range from 80 to 250°C at a rate 19 deg min<sup>-1</sup>, detector temperature 250°C, vaporizer temperature 250°C, column 2000 × 4 mm. IR spectra were recorded on spectrophotometer UR-20. Precise values of molecular ions mass were measured by high resolution mass spectroscopy on Finnigan MAT 8200 instrument. <sup>1</sup>H NMR spectra were registered on spectrometer Bruker WP-200SY, as internal reference were used residual protons in deuteriochloroform solvent ( $\delta$  7.24 ppm).

Fluorinated benzonitriles and tetrabutylammonium thiophenolate were prepared by published methods [16–20, 6]. The characteristics of all compounds obtained are consistent with the published data. As mediators were used anthracene, mp 216–217°C, 9,10-anthraquinone, mp 285–286°C, 4-cyanopyridine, mp 79–81°C, 4-nitrotoluene, mp 51–53°C, nitrobenzene, bp 85.4 ± 0.2°C/10 mm Hg; 9-nitroanthracene, mp. 146–148°C; bp 239°C (8 mm Hg) (cf. [21]).

**Electrochemical reduction of fluorinated benzonitriles.** Into an electrochemical cell was poured 0.1 M solution of TEAP in an aprotic solvent (7 ml into the cathode chamber and 4 ml into the anode chamber), into the cathode chamber was charged the required amount of fluorinated benzonitrile (*c* 0.05–0.1 M). The current was supplied to the cell at the voltage required for the electroreduction. The process was carried out at a constant potential, at room temperature, with stirring by a magnetic stirrer, and at constant argon stream through the cell. The reaction was monitored by intermittent recording of voltammograms of the reaction mixture. On completion of the reaction the reaction mixture from the cathode chamber was poured into 20 ml of water and depending on pH of the resulting mixture it was neutralized either with hydrochloric acid or sodium hydrogen carbonate. The reaction products were extracted into ethyl ether (5 × 7 ml) and dried with calcined magnesium sulfate. The mixture obtained on evaporation of ether was subjected to GLC analysis. At the use of mediator it

was placed into the cell together with the substrate, and the electroreduction was carried out at the reduction potential of the mediator. More detailed experimental conditions of reduction of benzonitriles under study are given in Table 1. For benzonitriles reduced with mediators were carried out control runs without mediators under the same experimental conditions. In all such control runs the final mixture contained only the initial nitrile.

**Electrochemical reduction of fluorinated benzonitriles in the presence of thiophenolate anion.** The electrochemical reduction of fluorinated benzonitriles in the presence of thiophenolate -anion was carried out as described above, but the 0.1 M TEAP solution was replaced by 0.1 M Bu<sub>4</sub>N<sup>+</sup>SPh<sup>-</sup> solution. The electrolysis conditions and the composition of reaction mixtures are given in more detail in Table 2. The electrolysis products were separated by TLC on silica gel-gypsum plate (silica gel of L100-160  $\mu$  grade), eluent chloroform.

**2-Fluorobenzonitrile.** The 2-phenylthiobenzonitrile obtained was purified by recrystallization from diluted methanol., bp 57–59°C (cf. [22]). IR and NMR spectra of compound obtained were fully in agreement with the assumed structure.

**2,4-Difluorobenzonitrile.** According to GC-MS data the reaction mixture contained alongside benzonitrile also compounds with molecular weight about twice as large as that of the initial nitrile, and a number of unidentified reaction products of minor content; the latter compounds were similar to those obtained in the absence of the nucleophile. No products of nucleophilic substitution were detected.

**2,4-Difluorobenzonitrile in the presence of mediator.** By means of TLC as described above were separated from the reaction mixture benzonitrile and the main fraction of mp 80–130°C that on recrystallization from hexane afforded colorless powder of mp 108–110°C.

According to the data of <sup>19</sup>F NMR spectroscopy the compound contained no fluorine. As found from mass spectrum:  $M^+$  319.0485; C<sub>19</sub>H<sub>13</sub>NS<sub>2</sub>. Calculated 319.049. Mass spectrum, *m/z* (*I*<sub>rel.</sub>, %) 319 (100); 210 (22.9); 209 (42.6), 183 (6.5); 139 (6.7), 109 (12.5); 77 (17.5); 65 (11.5); 51 (19.2). IR spectrum (in CHCl<sub>3</sub>),  $\nu$ , cm<sup>-1</sup>: 3070–3030, 1100–1050 (C<sub>ar</sub>-H); 2230 (C≡N);

1580, 1500-1450 (C=C<sub>ad</sub>). <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>), δ, ppm: 6.59 d (1H, CH<sub>ad</sub>, J<sub>H,H</sub> 1.8 Hz), 6.88 d.d (1H, CH<sub>ad</sub>, J<sub>H,H</sub> 1.8, 8.2 Hz), 7.40 d (1H, CH<sub>ad</sub>, J<sub>H,H</sub> 8.2 Hz), 7.1–7.5 m (10H, 10CH<sub>ad</sub>).

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