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ARYLATION OF ANIONS WITH DIARYLHALONIUM FLUOROBORATES
UNDER CONDITIONS OF INTERPHASE CATALYSIS

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Diarylhalonium salts are used as alkylating agents not only for theoretical investigations but also in preparative organic chemistry [1-3]. Arylation of anions by diarylhalonium salts, which is carried out under homogeneous conditions (water, aqueous alcohols, acetone, dioxane), requires as a rule extended time and heating since significant solvation of the arylhalonium cation and the reacting anion hinder the nucleophilic substitution at the C atom linked with the onium center.

We showed recently in [4] that phenylation of nitrite and toluene-p-sulfonate anions by diphenyliodonium fluoroborate may be carried out in a two-phase water-chloroform system. In this the reaction rate grew considerably in comparison with homogeneous media and the yield of the final product, too, in the case of the toluene-p-sulfonate anion (almost in two phases). The use of a special interphase catalyst was unnecessary in these reactions since the diphenyliodonium cation fulfilled this function [4].

The present work is devoted to a more detailed study of the arylation of anions by diaryliodonium, diphenylbromonium, and diphenylchloronium fluoroborates under conditions of interphase catalysis. The yields of liquid reaction products were determined by GLC (except for fluorobenzene, the identification and determination of which were effected with ^{19}F NMR spectroscopy). Crystalline products were isolated from reaction mixtures by preparative chromatography and were compared with authentic samples obtained by alternative synthesis.

The results of phenylation of the anions N_3^- , NO_2^- , SCN^- , and $\text{C}_6\text{H}_5\text{SO}_2^-$ by diphenyliodonium fluoroborate (I) in chloroform-water and dichloroethane-water systems are shown in Table 1, in which literature data on similar reactions in homogeneous media are included for demonstration. As is evident from Table 1, phenylation of anions N_3^- , NO_2^- , SCN^- , and PhSO_2^- by means of (I) in two-phase systems occurs under more mild conditions, significantly more rapidly than on using homogeneous media, and with high yields of the desired products.

The diphenyliodonium cation was poorly effective as an interphase catalyst in relation to certain anions and in such cases the reaction must be conducted using a special interphase

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TABLE 1. $\text{Ph}_2\text{IBF}_4^+ + \text{NaX} \rightarrow \text{PhI} + \text{PhX} + \text{NaBF}_4$

X	System	T., °C	Time, h	Yield of PhI, %	Substitution product		Literature reference
					formula	yield, %	
N ₃	CHCl ₃ /H ₂ O	56 ^a	0,5	96	PhN ₃	96	—
N ₃	Dioxane/H ₂ O	80	2	96-100	PhN ₃	97-99	[5]
NO ₂	CHCl ₃ /H ₂ O	56 ^a	1,5	98	PhNO ₂	99	—
NO ₂ ^b	H ₂ O	100	5	—	PhNO ₂	66	[6]
NO ₂	Dioxane/H ₂ O	100	50	77-79	PhNO ₂	72-76	[5]
SCN ^c	C ₂ H ₄ Cl ₂ /H ₂ O	72	10	90	PhSCN	90	—
					PhNCS	5	
SCN	Dioxane/H ₂ O	100	24	98-99	PhSCN	97-98	[5]
					PhNCS	0,28-0,3	—
PhSO ₂	CHCl ₃ /H ₂ O	56 ^a	2,5	99	Ph ₂ SO ₂	97	[6]
MeC ₆ H ₄ SO ₂ ^b	EtOH/H ₂ O	73	4	—	PhSO ₂ C ₆ H ₄ Me	56 ^d	

^aBoiling point of azeotropic mixture.

^bDiphenyliodonium bromide was put into the reaction.

^cReaction was carried out in a sealed glass ampul.

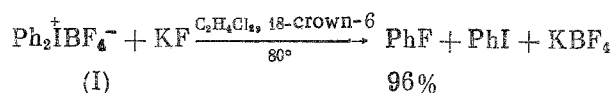
^dYield given was calculated on unreacted iodonium salt, 39% initial salt did not react.

TABLE 2. $\text{PhI}^+\text{ArBF}_4^- + \text{NaNO}_2 \xrightarrow[56^\circ]{\text{CHCl}_3/\text{H}_2\text{O}} \text{PhI} + \text{PhNO}_2 + \text{ArI} + \text{ArNO}_2 + \text{NaBF}_4$

Ar	Reaction time, h	Yield, %			
		PhI	PhNO ₂	ArI	ArNO ₂
2,4,6-Me ₃ C ₆ H ₂	0,25	100	0	0	98
4-NO ₂ C ₆ H ₄	1,0	93	1	1	98
Ph*	1,5	98	99	—	—
4-MeOC ₆ H ₄	3,0	8	90	90	9

*The experiment was carried out once again to complete the picture (see Table 1).

catalyst. For example, reaction of (I) with NaF in the systems CHCl₃-H₂O and C₂H₄Cl₂-H₂O went slowly, however, on boiling a mixture of (I), KF, and catalytic amounts of the ether 18-crown-6 in dichloroethane phenylation of the fluoride anion proceeded readily and fluorobenzene was formed in almost quantitative yield.



As is known, the substituents in the aromatic ring exert a large influence on the reactivity of iodonium salts and on the direction of their reaction with nucleophiles. Electron-accepting substituents activate iodonium salts to reactions of the S_NAr type, electron-donating substituents passivate [7, 8]. It is also known that the presence of ortho substituents in the aryl ligand strongly facilitate nucleophilic substitution in this ligand (so-called ortho-effect) [9, 10]. If the ortho substituent is absent, then attack of the nucleophile is directed predominantly toward that radical of those bound to the iodonium centers which is the most electron deficient.

We decided to check whether these regularities were preserved when carrying out reactions of iodonium salts under conditions of interphase catalysis. The following unsymmetrical diaryliodonium salts were chosen as subjects of the investigation, viz., the fluoroborates of 4-nitrodiphenyliodonium (containing a strong electron-donating substituent), and diphenylmesityliodonium (with a sterically hindered ligand). Reactions of these compounds with NaNO₂ were carried out in a chloroform-water system. The results of experiments are shown in Table 2, from which it follows that all the regularities stated above were retained on going over from homogeneous media to the CHCl₃-H₂O system. In view of the abundance of literature data on nucleophilic substitution reactions in unsymmetrical diaryliodonium salts, we limit ourselves to refer only to the literature review in dissertation [11] devoted to this problem.

TABLE 3. $\text{Ph}_2\text{HalBF}_4^+ + \text{NaSCN} \xrightarrow[72^\circ]{\text{C}_2\text{H}_5\text{Cl}_2/\text{H}_2\text{O}} \text{PhSCN} + \text{PhNCS} + \text{PhHal} + \text{NaBF}_4$

Halonium salt	Reaction time, h	Yield of phenylation products PhSCN + PhNCS, %
$\text{Ph}_2\text{IBF}_4^+$ †	10	95
$\text{Ph}_2\text{BrBF}_4^+$	0,5	95
$\text{Ph}_2\text{ClBF}_4^+$	0,25	93

*In all cases the yield of PhNCS was 2-5%.

†The experiment was carried out once again to complete the picture (see Table 1).

TABLE 4. Reactions of Diphenylhalonium Fluoroborates with Sodium Benzenesulfinate in a $\text{CHCl}_3\text{-H}_2\text{O}$ System at 20°C

Cation	Reaction time, h	Yield of diphenylsulfone, %	Other products
Ph_2I^+	64	99	PhI
Ph_2Br^+	7	77	PhBr, PhS(O)OPh, PhOH
Ph_2Cl^+	5	68	PhCl, PhS(O)OPh*, PhOH

*The yield of phenyl benzenesulfinate isolated preparatively was 12%.

Like diaryliodonium salts, bromonium and chloronium compounds of the aromatic series are also able to arylate nucleophiles [3]. In homogeneous media bromonium and chloronium salts are more reactive in relation to nucleophilic reagents than their iodonium analogs. In the present work we studied the reaction of the fluoroborates of diphenylbromonium (II) and diphenylchloronium (III) with the anions SCN^- and PhSO_2^- under conditions of interphase catalysis and compared their behavior with (I) in these reactions. As is seen from Table 3, (II) and (III) like (I) readily phenylated SCN^- while the time needed to complete these reactions was in complete accord with generally known concepts on the reactivity of iodonium, bromonium, and chloronium compounds.

It should be mentioned that under conditions of interphase catalysis reactions of (I)-(III) with NaSCN led to phenyl isothiocyanate in 2-5% yield while on phenylation of the SCN^- anion by means of (I) in aqueous dioxane the yield of PhNCS was more than an order of magnitude less and was 0.28-0.3% generally [5]. This is probably connected with the fact that, under conditions of interphase catalysis, the reaction mainly proceeds in a far less solvating medium than water. The N atom in the SCN^- anion carried the greatest negative charge [12] and, consequently, in aqueous solution where solvation is explained mainly by orientation interactions between particles it will be most strongly hydrated. This hinders attack by the diphenyliodonium cation at the nitrogen atom of SCN^- and leads to a very low yield of N-phenylated product. In the low polarity organic phase solvation of the anion is on the whole incomparably weaker and has a somewhat different character (here the contribution of the dispersion interactions between the anion and the solvent molecules is greater). In the character of the interactions the density of the solvation "fur-coat" is displaced somewhat from the less polarizable nitrogen atom to the more polarizable sulfur atom. As a result of this, attack at the nitrogen atom is attenuated to a somewhat greater extent than attack at the sulfur atom and the yield of phenyl isothiocyanate grows.

Particularly interesting results were obtained on carrying out reactions of (I)-(III) with PhSO_2Na in a $\text{CHCl}_3\text{-H}_2\text{O}$ system at about 20°C (Table 4).

As in the case of the rhodanide anion, the rate of reaction of diphenylhalonium cations with PhSO_2Na increased in the series $\text{Ph}_2\text{I}^+ < \text{Ph}_2\text{Br}^+ < \text{Ph}_2\text{Cl}^+$. The fact discovered by us of the reduction in yield of diphenylsulfone along the same series was far more interesting. The yield of benzenesulfonic acid phenyl ester and phenol increased in the same sequence in parallel, i.e., as for the products formed by O-phenylation of the PhSO_2^- anion. The phenyl ester of benzenesulfonic acid was readily hydrolyzed; consequently its precise yield may not be indicated. As a result of some difference in the reaction rates of Ph_2Hal^+ with PhSO_2^- and hydrolysis of the resulting benzenesulfonic acid ester we failed to isolate a certain amount of this substance by preparative chromatography and to show its identity with the phenyl ester of benzenesulfonic acid obtained by an alternative synthesis in [13]. Phenol is evidently formed as a result of hydrolysis of phenyl benzenesulfinate although it is not

excluded that a small amount of it may also be formed on interaction of the halonium salt with hydroxyl anion.*

The growth of the yield of O-phenylation products of benzenesulfinate anion by diphenylhalonium salts in the series $\text{Ph}_2\overset{+}{\text{I}} < \text{Ph}_2\overset{+}{\text{Br}} < \text{Ph}_2\overset{+}{\text{Cl}}$ may be explained by an increase in the same series of the contribution of the electrostatic term (determining the progress of the charge-controlled reaction) to a value changing the overall energy on donor-acceptor interaction [12]. The absolute value of the electrostatic term was proportional to the size of the charges on the coordinated donor and acceptor atoms and since the positive charge on the C atom linked with the onium centers is increased [14] in the series $\text{Ph}_2\overset{+}{\text{I}} < \text{Ph}_2\overset{+}{\text{Br}} < \text{Ph}_2\overset{+}{\text{Cl}}$ the inclination of the cations to enter a charge-controlled reaction must grow in the same series. Since the benzenesulfinate anion has a tendency to attack the electrophile with the O atom bearing the greatest negative charge in a charge-controlled reaction, an understandable reason remains for the increase in yield of O-phenylated product along the halonium cation series indicated above. In the terms of the Pearson principle on the hardness and softness of acids and bases the hardness of the C atom linked to the onium center is increased from the diphenyliodonium cation to the diphenylchloronium, which leads to easing of the attack of this atom by the hard oxygen of the benzenesulfinate anion.

It should be mentioned that on interaction of diphenylhalonium cations with ambident anions in two-phase systems the ratio of arylation products at the harder and softer centers will be changed unambiguously depending on the nature of the halogen in not every case. For example, in reactions with rhodanide ion this was not observed. The fact is in two-phase systems of water-organic solvent the reactions of halonium salts proceed mainly in the organic phase and certainly partly in water also (reaction was also possible at the separation boundary of the phases). The ratio of the arylation products of an ambident anion by any halonium compound in water or in low polarity organic phase may be completely different, since the contributions of the charge and orbitally controlled reactions in a general donor-acceptor interaction depend on the dielectric constant of the solvent [12]. Consequently, if the more reactive diphenylhalonium cations (chloronium, bromonium) possibly improve reaction with certain anions to some extent in water up to the level as occurs with interphase transfer and reaction in the organic phase, then this, of course, may influence significantly the ratio of phenylation products at the harder and softer centers.

EXPERIMENTAL

GLC was carried out on a Khrom-4 chromatograph with a Kent Chromalog-2 electronic integrator. Glass columns were packed with Chromosorb W 80/100 mesh coated with Dexil-200 (2%). Insertion of samples was effected directly onto the column. The temperature program was from 80 \rightarrow 250°C at a rate of 6 deg/min. Carrier gas (He) flow rate was 30 ml/min. Chlorobenzene was used as internal standard in GLC analyses [except for the reaction of (III) with NaSCN in which iodobenzene was taken]. Preparative chromatography was carried out on columns of quartz glass with silica gel 5/40 μm with a luminescent indicator on detection in UV light from a Foton instrument. ^{19}F NMR spectra were obtained on a Bruker WP-200 SY instrument (operating frequency 188.31 MHz), chemical shifts are given on the δ scale relative to $\text{CF}_3\cdot\text{CO}_2\text{H}$ as external standard.

The fluoroborates of diphenyliodonium and diphenylbromonium, and the bromide of 4-methoxydiphenyliodonium were synthesized by the methods of [15-17], respectively.

Diphenylchloronium Fluoroborate (III). A solution of diphenyldiazonium fluoroborate (9.89 g) in CF_3COOH (15 ml) was added dropwise to a boiling mixture of chlorobenzene (30 ml) and CF_3COOH (30 ml) and the mixture was boiled until a negative test with β -naphthol. The solution was evaporated to minimum volume and (III) was precipitated by adding ether to the residue which was then reprecipitated twice with ether from acetone. Yield was 1.55 g (11%), mp 118°C (cf. [18]). Found, %: C 52.42; H 3.65. $\text{C}_{12}\text{H}_{10}\text{BClF}_4$. Calculated, %: C 52.13; H 3.65.

Phenylmesityliodonium Fluoroborate. Concentrated H_2SO_4 (10 ml) was added with stirring to a solution of phenyliodosodiacetate [19] (11 g) and mesitylene (4.2 ml) in Ac_2O (150 ml) cooled to 5°C, at such a rate that the temperature remained below 10°C. The mixture was stirred for 2 h, poured onto ice (200 g), 40% HBF_4 (50 ml) was added, and the mixture was extracted with chloroform to a negative test of the aqueous layer with NaI. The combined ex-

*An aqueous solution of PhSO_2Na has a weakly alkaline reaction.

tracts were filtered through a layer of Al_2O_3 and evaporated to minimum volume. Phenylmesityliodonium fluoroborate (9.6 g: 63%) was precipitated by adding ether to the residue and had mp 195-195.5°C (after two reprecipitations with ether from acetone). Found, %: C 43.80; H 3.93. $\text{C}_{15}\text{H}_{16}\text{BF}_4\text{I}$. Calculated, %: C 43.94; H 3.93.

4-Methoxydiphenyliodonium Fluoroborate. 4-Methoxydiphenyliodonium bromide (3 g) was dissolved in a solution of triethoxonium fluoroborate [20] (9 g) in dry acetone (70 ml), then alcohol (10 ml) was added, and solvents were removed in vacuum. By adding ether to the residue 4-methoxydiphenyliodonium fluoroborate was precipitated, which was then passed in chloroform through a layer of Al_2O_3 and was reprecipitated three times with ether from acetone. Yield was 2.15 g (72%) of mp 99°C (cf. [8]).

4-Nitrodiphenyliodonium Fluoroborate. Concentrated H_2SO_4 (130 ml) was added carefully at -10 to +15°C to a stirred solution of p-nitroiodobenzene (4.61 g) in Ac_2O (70 ml) and then benzene (25 ml) and finely powdered $\text{K}_2\text{S}_2\text{O}_8$ (14 g) were added at -10°C. The mixture was stirred for 1.5 h at -10°C, then for 6 h at 0-5°C, and 10 h at 20°C, after which it was poured onto ice (300 g), filtered, and washed with benzene. 4-Nitrodiphenyliodonium iodide (4.55 g: 54%) was precipitated by the action of KI on the obtained solution and had mp 127-128°C (with decomposition, cf. [21]). 4-Nitrodiphenyliodonium iodide (4.55 g), water (30 ml), and Ag_2O (1.3 g) were carefully ground in a mortar, filtered, and the filtrate treated with 40% HBF_4 to acid reaction. The acid solution was extracted with nitromethane to a negative test with NaI. Nitromethane was removed in vacuum, the residue passed in acetone through a layer of Al_2O_3 , and three times reprecipitated with ether from acetone. 4-Nitrodiphenyliodonium fluoroborate (2 g: 48%) of mp 155-157°C was obtained. Found, %: C 35.12; H 2.09; N 3.86. $\text{C}_{12}\text{H}_9\text{BF}_4\text{NO}_2\text{I}$. Calculated, %: C 35.06; H 2.21; N 3.41.

General Procedure for Carrying Out the Reaction of (I) with NaN_3 , NaNO_2 , and NaSCN . A mixture of (I) (1 g: 2.72 mmole) Na salt (13.6 mmole), organic solvent (5 ml), and water (5 ml) was stirred vigorously in a flask with a reflux condenser until complete decomposition of (I). The end of the reaction was determined by the absence of a precipitate in a test sample of aqueous phase on adding a solution of NaI. A precisely measured amount of internal standard was added to the organic phase and the mixture was investigated by GLC.

General Procedure for Carrying Out the Reaction of (II) and (III) with NaSCN . Substance (II) or (III) (0.36 mmole), NaSCN (1.85 mmole), dichloroethane (1 ml), and water (1 ml) were stirred vigorously at the boiling point in a flask with a reflux condenser until a test sample of the aqueous layer did not give a precipitate with a solution of NaHgBr_3 . The internal standard was added to the organic phase and the mixture investigated by GLC.

Reaction of (I) with PhSO_2Na at 56°C. A mixture of (I) (1 g), PhSO_2Na (2.23 g), water (5 ml), and CHCl_3 (5 ml) was stirred vigorously at the boiling point until complete decomposition of the iodonium salt. The organic layer was filtered through Al_2O_3 , the solvent distilled off, and the residue washed with cold pentane. Yield of diphenylsulfone was 0.57 g (97%) of mp 123-124°C (cf. [22]).

General Procedure for Carrying Out the Reactions of (I), (II), and (III) with PhSO_2Na at 20°C. A mixture of (I), (II), or (III) (1.8 mmole), PhSO_2Na (9 mmole), CHCl_3 (5 ml), and H_2O (5 ml) was stirred at 20°C until complete decomposition of the halonium salt. The organic layer was evaporated, and diphenylsulfone, phenyl benzenesulfinate, and phenol were isolated from the residue by column chromatography in the system chloroform-pentane (3:2).

Reaction of Phenylmesityliodonium Fluoroborate with NaNO_2 . A mixture of phenylmesityliodonium fluoroborate (1 g), NaNO_2 (0.85 g), CHCl_3 (5 ml), and water (5 ml) was stirred at the boiling point until decomposition of the iodonium salt. Internal standard was added to the mixture, the organic layer was separated, investigated by GLC, and then evaporated. The residue was chromatographed on a column. The first fraction (iodobenzene) was eluted with pentane and a second (nitromesitylene) with chloroform. Nitromesitylene (0.396 g: 98%) was isolated having mp 39-41°C (cf. [22]).

Reaction 4-Nitrodiphenyliodonium Fluoroborate with NaNO_2 . A mixture of 4-nitrodiphenyliodonium fluoroborate (0.565 g), NaNO_2 (0.47 g), CHCl_3 (5 ml), and water (5 ml) was stirred at the boiling point until complete decomposition of the iodonium salt. Internal standard was added to the mixture, the organic layer was investigated by GLC, and then evaporated. p-Dinitrobenzene (0.225 g: 98%) of mp 173°C (cf. [22]) and p-nitroiodobenzene (0.004 g: 1%) of mp 170-171°C (cf. [22]) were isolated from the residue by column chromatography in the system chloroform-petroleum ether (1:6).

Reaction of 4-Methoxydiphenyliodonium Fluoroborate with NaNO₂. Under the conditions of the previous experiment p-iodoanisole (0.500 g: 90%) of mp 50°C (cf. [22]) and p-nitroanisole (0.033 g: 9%) of mp 54°C (cf. [22]) were isolated from 4-methoxydiphenyliodonium fluoroborate (0.938 g) and NaNO₂ (0.82 g) by column chromatography (chloroform-pentane, 1:3).

Reaction of (I) with KF. A mixture of freshly roasted KF (0.286 g), (I) (0.350 g), dichloroethane (2 ml), and 18-crown-6 ether (0.05 g) was heated at 80°C in a sealed ampul with vigorous agitation for 12 h. Benzotrifluoride (0.105 g) was added to the mixture and the ¹⁹F NMR spectrum of the liquid phase was measured. Three signals were observed in the spectrum, at -12.7 ppm (CF₃Ph), +38.1 ppm (PhF), and -76.8 ppm (BF₄⁻). It followed from the integrated intensities of the signals for CF₃Ph (3.3) and PhF (1.4) that the yield of the latter was 96%.

CONCLUSIONS

1. Arylation of anions by diarylhalonium fluoroborates under conditions of interphase catalysis proceeds more rapidly than in homogeneous media and as a rule in higher yield of the desired products.

2. The rules for the influence of substituents in the aromatic ligands of the iodonium salts on their reactivity are retained on going over from homogeneous media to a two-phase system.

3. The yield of O-phenylation products grew in the series Ph₂I⁺ < Ph₂Br⁺ < Ph₂Cl⁺ in the reaction of diphenylhalonium cations with the benzenesulfinate anion.

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