rate constant ($k_1^{obsd}$ or $k_0^{obsd}$) in Tables I and II is the average of 3–5 runs.

Each pseudo-first-order rate constant, $k_1^{obsd}$ (Table I), was converted to a second-order rate constant, taking into account the substrate concentration $[1]_s$ and the difference between the actual bromine concentration, $[Br_2]$, and the stoichiometric concentration of bromine, $[Br_2]_s$.[12]

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# Aryl Radical Departure Aptitudes in Reactions of Diaryliodonium Fluoroborates with Sodium Ethoxide

Jacek J. Lubinkowski,* Cecilia Gimênez Arrieche, and William E. McEwen*

*Departments of Chemistry, Universidad Simon Bolivar, Apartado 80659, Caracas, Venezuela, and The University of Massachusetts, Amherst, Massachusetts 01003*

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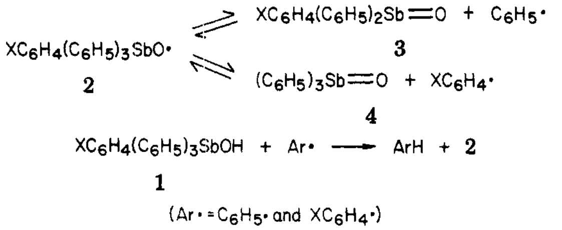
Several unsymmetrical diaryliodonium fluoroborates have been prepared and subjected to reaction with sodium ethoxide in ethanol solution at 71 °C. From quantitative determinations of products, it has been possible to calculate apparent departure aptitudes of several common aryl radicals from radical cations of type **6**. These results are compared with similar data obtained from thermolysis reactions of hydroxytetraarylstiboranes.

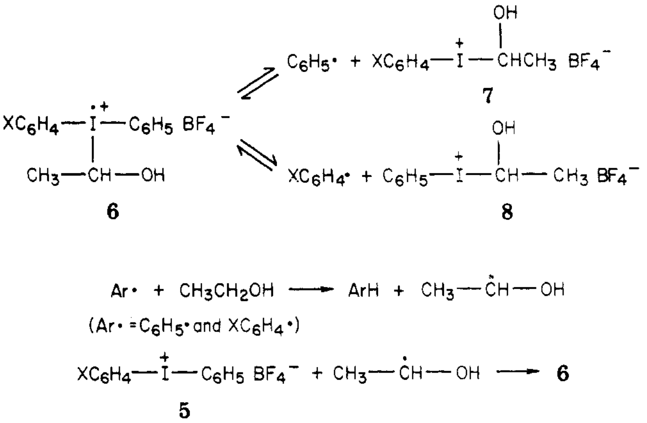
Hydroxytetraarylstiboranes of the type $XC_6H_4(C_6H_5)_3SbOH$ (**1**) have been reported[1-4] to undergo thermolysis at 30–50 °C by a radical chain mechanism. Evidence has been presented that the following competitive reactions occur in the propagation stage of each decomposition:



Since material balances were realized in these reactions, the ratios $3C_6H_5X/C_6H_6$ could be used as a measure of the apparent relative departure aptitudes of the various aryl radicals from **2**. These departure aptitudes, corresponding to each of the substituents X in **1**, were determined to be as follows: X = $p$-$NO_2$, 5.6; $m$-$NO_2$, 9.4; $p$-Cl, 2.5; $m$-Cl, 4.6; $p$-$CH_3$, 1.3; $m$-$CH_3$, 0.78; $p$-$OCH_3$, 2.6; $m$-$OCH_3$, 2.5.

A plot of log (departure aptitude) vs. $\sigma^\circ$ was found to be linear for the meta-substituted aryl groups; $\rho = 1.40$, correlation coefficient = 0.94. The $\sigma^\circ$ values are derived from reactions of compounds in which the aromatic ring is shielded from the reaction center (e.g., ionization of phenylacetic acids) or where the substituent is located in the meta position only. Taft[5] believes that these values are the best for use where only nonconjugative effects are involved. Most of the departure aptitudes for the para-substituted aryl groups cannot be correlated by use of the Hammett equation utilizing $\sigma^\circ$ values, and this suggests that there is a conjugative interaction of some type between the para substituent and the odd electron. This conjugative effect apparently causes an incremental decrease in stability of the radical when a strongly electron-withdrawing substituent (e.g., $p$-$NO_2$) is present, but an incremental increase in stability when an electron-donating substituent (e.g., $p$-MeO or $p$-Me) is present. For example, the departure aptitude of the $p$-methoxyphenyl group would be 0.76 based on the plot and the $\sigma^\circ$ value of the $p$-MeO group. Thus, the observed departure aptitude of the $p$-methoxyphenyl radical is apparently 3.4-fold greater than that expected if no conjugative influence were operative. In like manner, the departure aptitude of the $p$-methylphenyl radical would be 0.71 based on the plot and the $\sigma^\circ$ value of the $p$-Me group, and the observed departure aptitude of the $p$-methylphenyl group is 1.8-fold greater than that expected if no conjugative interaction were operative.

Scheme I



The reaction of a diaryliodonium salt with a metal alkoxide to give an alkyl aryl ether plus an aryl iodide is considered to be of synthetic utility; however, an aromatic hydrocarbon, an aryl iodide, and an aldol resin (or a ketone if the alkoxide is derived from a secondary alcohol) are frequently the major products. A mechanism which has been proposed for the formation of the latter set of

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products involves a radical chain reaction which is reminiscent of that proposed for the thermolysis of **1**. The competitive propagation steps for the reaction of $XC_6H_4I^+C_6H_5BF_4^-$ (**5**) with sodium ethoxide in ethanol, for example, are thought to be as shown in Scheme I.

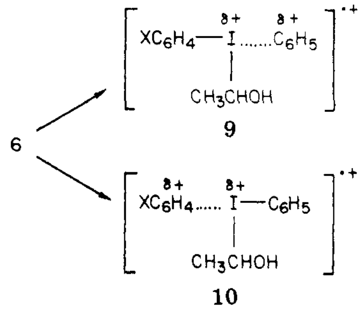
The ratios $C_6H_5X/C_6H$ in these reactions would reflect the relative departure aptitudes of the various aryl radicals from **6**, and it was of interest to us to find out whether there was any logical relationship between these values and the ones obtained from the data on the thermolysis of **1**. The reactions of four unsymmetrical diaryliodonium fluoroborates with sodium ethoxide in ethanol solution at 71 °C for 3 h have now been studied in a quantitative manner, and the apparent departure aptitudes, corresponding to each of the substituents X in **5**, were determined to be as follows: X = *m*-$NO_2$, 5.6; *m*-$CH_3$, 1.2; *p*-$CH_3$, 1.1; *p*-$OCH_3$, 0.8.

Although the data are limited, a plot of log departure aptitude for the meta-substituted aryl groups (*m*-$NO_2$, *m*-Me, H) vs. $\sigma^\circ$ is linear with $\rho = 0.92$ and correlation coefficient = 0.96.

The key to a rationalization of the results consists of the fact that, whereas **2** is a neutral radical which gives rise to electrically neutral products ($C_6H_5\cdot$, $XC_6H_4\cdot$, **3**, and **4**), **6** is a radical cation which gives rise to neutral aryl radicals plus the new cations **7** and **8**. Clearly, the effects of the substituents X on the relative stabilities of the aryl radicals $XC_6H_4\cdot$ are important but so also are the effects of X on the relative stabilities of the triarylstibine oxides **3** and **4** and on the cations of **7** and **8**. With respect to electron-donating substituents, at least, the stabilizing effects of the cations of **7** and **8** would be distinctly greater than those on the electrically neutral stibine oxides **3** and **4**. Thus, since the ease of dissociation of **2** and **6** depends on the relative stabilities of both fragments formed in each dissociation, the effect of an electron-donating substituent (X) will be to create a bias in favor of the formation of a larger relative amount of **7** plus $C_6H_5\cdot$ from **6** than of **3** plus $C_6H_5\cdot$ from **2**. In this context, the change in the apparent relative departure aptitude of the *p*-methoxyphenyl radical from a value of 2.6 in the decomposition of **1** to a value of 0.8 in the reaction of **5** with sodium ethoxide is readily understandable; the strongly electron-donating *p*-methoxy group aids the formation of **7** more than it aids the formation of **3**, and this results in a greater relative amount of $C_6H_5\cdot$ being formed from **6** than from **2**. Similar, but smaller, effects are observed when X = *p*-$CH_3$. In other words, the greater stabilizing effect of the *p*-MeO group on **7** vs. **3** would counteract the greater ease of formation of the *p*-methoxyphenyl radical vs. that of the phenyl radical, and the calculated value of the departure aptitude of the *p*-methoxyphenyl group based on the plot for the iodonium salt reaction and the $\sigma^\circ$ value of the *p*-MeO group would be much closer to the observed value. In fact, the calculated value turns out to be 0.85 compared to the observed value of 0.80. Since the *p*-Me group is not as strong an electron-donor group as *p*-MeO, the "correction" in the former case would be less. The calculated departure aptitude for the *p*-methylphenyl group based on the plot and the $\sigma$ value of *p*-Me turns out to be 0.80 compared to the observed value of 1.1.

On a more fundamental basis, the phenyl radical has been shown to be a $\sigma$ radical,[9] with bond bending bringing about a competition for the odd electron between a localized p orbital and a delocalized p orbital.[10] Furthermore, the presence of substituents on the phenyl radical can lead to an apparent increase in delocalization as reflected in the increase in spin density found at the other carbon atoms of the ring.[11] With regard to the positive $\rho$ values for the Hammett plots discussed previously, this is attributable mainly to the differences in bond-dissociation energies involved in the competitive decompositions of **2** and **6**. For example, the $D_{CN}$ values reported for thermolysis of nitrobenzene, *m*-dinitrobenzene, and *m*-nitrotoluene are 70.4, 66.1, and 70.7 kcal/mol, respectively,[12] which clearly dictates a positive value of $\rho$ in Hammett correlations of departure aptitudes of aryl groups in such radical dissociations.

However, Pryor and co-workers[13] have pointed out that the $\rho$ values for Hammett plots of radical reactions depend not only on the effects of substitution on the bond-dissociation energies (BDE) of the reactants but also on the effects of substituents on the stabilities of the transition states (SETS) of the reactions. In the dissociation of **2** and **6**, the BDE values are mainly responsible for the positive values of $\rho$, as stated above. However, whereas the modification of $\rho$ by SETS in the case of **2** would be small because of the fact that **2** is electrically neutral, the modification of $\rho$ by SETS in the case of **6** assumes greater importance. The positive charge of **6** is reflected in the transition states of the competitive dissociation processes as shown in **9** and **10**. When X is electron withdrawing,



this will tend to raise the energy of **10** relative to **9**, and when X is electron donating, the reverse effect will be operative. Thus, the perturbation due to SETS would tend to make the value of $\rho$ less positive in the dissociation of **6**, which is in accord with the facts.

The four unsymmetrical diaryliodonium salts which were utilized to obtain the data cited above were phenyl-*p*-methoxyphenyliodonium fluoroborate (**5**, X = *p*-$OCH_3$),[14] phenyl-*p*-tolyliodonium fluoroborate (**5**, X = *p*-$CH_3$),[15] phenyl-*m*-tolyliodonium fluoroborate (**5**, X = *m*-$CH_3$),[16] and *p*-tolyl-*m*-nitrophenyliodonium fluoroborate.[17]

Detailed data for the reaction of phenyl-*p*-tolyliodonium fluoroborate with sodium ethoxide in ethanol at 71 °C has been provided in our previous paper.[7] The results of the corresponding reaction of phenyl-*p*-methoxyphenyl-

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(17) This compound was obtained by heating an aqueous mixture of fluoroboric acid, toluene, and *m*-nitroiodobenzene diacetate; the latter compound was prepared by the method of Willgerodt, C. *Chem. Ber.* **1893**, *26*, 1312.

Table I. Reaction of Phenyl-*p*-methoxyphenyliodonium Fluoroborate with Sodium Ethoxide[a]

| expt | % yields of products | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | PhH | PhOMe | PhOEt | PhI | *p*-MeO-PhOEt | *p*-IPhOMe | PhPh | PhPh-OMe-*p* | *p*-MeOPh-PhOMe-*p* |
| 1 | 26.9 | 21.9 | 14.6 | 24.3 | 1.2 | 55.0 | *b* | *b* | *b* |
| 2 | 26.9 | 22.3 | 14.8 | 23.4 | 1.2 | 55.6 | *b* | *b* | *b* |
| 3 | 28.0 | 23.5 | 15.8 | 24.3 | 1.2 | 49.8 | *b* | *b* | *b* |
| 4 | 27.1 | 23.3 | 15.8 | 24.3 | 1.2 | 49.8 | *b* | *b* | *b* |

[a] Each reaction was carried out with 5 × $10^{-4}$ mol of the iodonium salt and 7 × $10^{-4}$ mol of sodium ethoxide at 71.0 ± 0.5 °C for 3 h in air. [b] Less than 0.01% as determined by VPC.

Table II. Reaction of Phenyl-*m*-tolyliodonium Fluoroborate with Sodium Ethoxide[a]

| expt | % yields of products | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | PhH | PhMe | PhOEt | PhI | *m*-IPhMe | *m*-MePhOEt | PhPh | PhPh-Me-*m* | *m*-MePh-PhMe-*m* |
| 1 | 19.8 | 22.6 | 12.0 | 37.3 | 40.9 | 9.4 | *b* | *b* | *b* |
| 2 | 20.1 | 22.3 | 11.9 | 37.6 | 39.9 | 9.2 | *b* | *b* | *b* |
| 3 | 20.1 | 24.1 | 13.7 | 35.7 | 44.0 | 10.0 | *b* | *b* | *b* |
| 4 | 20.3 | 24.2 | 14.6 | 36.3 | 44.9 | 10.5 | *b* | *b* | *b* |

[a] Each reaction was carried out with 5 × $10^{-4}$ mol of the iodonium salt and 7 × $10^{-4}$ mol of sodium ethoxide at 71.0 ± 0.5 °C for 3 h in air. [b] Less than 0.01% as determined by VPC.

Table III. Reaction of *p*-Tolyl-*m*-nitrophenyliodonium Fluoroborate with Sodium Ethoxide[a]

| expt | % yields of products | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | PhMe | $PhNO_2$ | *p*-MePhOEt | *p*-MePhI | *m*-$NO_2$PhOEt | *m*-$NO_2$-PhI | *p*-MePh-PhMe-*p* | *p*-MePh-$PhNO_2$-*m* | *m*-$NO_2$Ph-$PhNO_2$-*m* |
| 1 | 0.9 | 4.7 | 3.7 | 80.4 | 76.8 | 3.4 | *b* | *b* | *b* |
| 2 | 0.9 | 4.7 | 3.8 | 79.0 | 80.3 | 3.6 | *b* | *b* | *b* |
| 3 | 0.9 | 4.7 | 3.8 | 80.3 | 79.8 | 3.2 | *b* | *b* | *b* |
| 4 | 1.0 | 4.8 | 3.8 | 80.6 | 79.8 | 3.3 | *b* | *b* | *b* |

[a] Each reaction was carried out with 5 × $10^{-4}$ mol of the iodonium salt and 7 × $10^{-4}$ mol of sodium ethoxide at 71.0 ± 0.5 °C for 3 h in air. [b] Less than 0.01% as determined by VPC.

iodonium fluoroborate are provided in Table I. The average yield of anisole, based on four experiments, was 22.75 ± 0.65%, while the yield of benzene was 27.23 ± 0.39%. The results of the corresponding reaction of phenyl-*m*-tolyliodonium fluoroborate are given in Table II. The average yield of toluene, based on four experiments, was 23.30 ± 0.85%, while the yield of benzene was 20.08 ± 0.07%. The results of the reaction of *p*-tolyl-*m*-nitrophenyliodonium fluoroborate with sodium ethoxide are provided in Table III. The average yield of nitrobenzene was 4.73 ± 0.04%, while that of toluene was 0.93 ± 0.04%.

We have previously suggested[8] that the transition state for the $S_NAr$ reaction of a diaryliodonium salt with a base is an early one, a significant degree of negative charge remaining on the attacking nucleophile, thus permitting a favorable electrostatic interaction with the positively charged iodine. Of course, the presence of a substituent also influences the energy of the transition state, and, in an unsymmetrical diaryliodonium salt, attack of the nucleophile will predominate on that ring which bears a more electron-withdrawing substituent. Furthermore, when an electron-withdrawing substituent is present in the diaryliodonium cation, the yields of the products of $S_NAr$ reactions increase at the expense of the products of the radical chain reactions. Thus, in the reaction of *p*-tolyl-*m*-nitrophenyliodonium fluoroborate with sodium ethoxide, it is not surprising that *m*-nitrophenetole is obtained in 79.2 ± 1.2% yield, *p*-methylphenetole in 3.8 ± 0.1% yield, and the products of the radical chain reaction, nitrobenzene and toluene, in but 4.73 ± 0.04 and 0.93 ± 0.04% yield, respectively.

Conversely, in the reaction of phenyl-*p*-methoxyphenyliodonium fluoroborate with sodium ethoxide, the presence of the electron-donating methoxy group causes the overall yield of the products of $S_NAr$ reactions to decrease, but, as expected, phenetole is obtained in higher yield (15.3 ± 0.6%) than *p*-methoxyphenetole (1.2 ± 0.1%). In like manner, the presence of the mildly electron-donating methyl group in phenyl-*m*-tolyliodonium fluoroborate causes the yields of phenetole and *m*-methylphenetole to be but 13.1 ± 1.1 and 9.8 ± 0.5%, respectively. As reported previously,[7] the same situation holds true for the reaction of phenyl-*p*-tolyliodonium fluoroborate with sodium ethoxide; the yield of phenetole is 9.5% and that of *p*-methylphenetole 2.6%.

## Experimental Section

**Phenyl-*m*-tolyliodonium Bromide.** This compound was prepared according to the method of Caserio, Glusker, and Roberts[16] from 10.6 g (0.045 mol) of *m*-iodosotoluene. The product weighed 5.0 g (33%), mp 180–182 °C (lit.[16] mp 182.5–183.5 °C)

**Phenyl-*m*-tolyliodonium Fluoroborate.** Freshly prepared silver oxide (1.5 g, 6.5 mmol) was dissolved in a 20-mL aqueous solution containing 5.0 g of 50% fluoroboric acid. The solution was brought to the boiling point, and 3.9 g (0.011 mol) of solid phenyl-*m*-tolyliodonium bromide was added. After the reaction mixture had been refluxed for 30 min, the excess silver oxide and the silver bromide which had formed were removed by filtration, and the filtrate was allowed to cool in the refrigerator overnight. The precipitate which formed was collected and recrystallized from a minimal amount of water. The yield was 1.60 g (39%): mp 123.5–125 °C; IR (KBr) 3050, 1600, 1550, 1470, 1450, 1050, 990, 780, 750, 650 $cm^{-1}$; NMR ($CDCl_3$) $\delta$ 2.50 (s, 3 H), 7.7 (m, 6 H), 8.3 (m, 3 H).

Anal. Calcd for $C_{13}H_{12}IBF_4$: C, 40.88; H, 3.17. Found: C, 40.67; H, 3.13.

**Phenyl-*p*-methoxyphenyliodonium Bromide.** This compound was prepared according to the method of Beringer[14] from

25 g (0.11 mol) of iodosobenzene. The product weighed 28.8 g (70%), mp 183–184 °C dec (lit.[14] mp 185 °C dec).

**Phenyl-*p*-methoxyphenyliodonium Fluoroborate.** This compound was prepared according to the method of Berlinger[14] from 4.0 g (0.010 mol) of phenyl-*p*-methoxyphenyliodonium bromide. The product weighed 3.4 g (74%), mp 91–94 °C (lit.[14] mp 91–94 °C).

***p*-Tolyl-*m*-nitrophenyliodonium Fluoroborate.** A slurry of 3.0 g (8.2 mmol) of *m*-nitroiodobenzene diacetate[17] in 1.5 g (16.3 mmol) of toluene and 1.5 g (17 mmol) of 50% fluoroboric acid was heated at 50 °C for 12 h with vigorous stirring. The white precipitate which formed was collected by filtration and washed with ether, yielding 2.4 g (69%) of the desired product, which was recrystallized from 95% ethanol: mp 166–167 °C; IR (KBr) 3100, 2740, 1600, 1530, 1350, 1050, 1000, 800, 735 cm$^{-1}$; NMR (Me$_2$SO) $\delta$ 2.33 (s, 3 H), 7.3 (m, 2 H), 7.75 (t, 1 H, $J$ = 8.0 Hz), 8.2 (m, 2 H), 8.5 (m, 2 H), 9.1 (t, 1 H, $J$ = 2.3 Hz).

Anal. Calcd for $C_{13}H_{11}INO_2BF_4$: C, 36.57; H, 2.59; N, 3.28. Found: C, 36.36; H, 2.61; N, 3.23.

***m*-Methylphenetole.** This compound was prepared according to the method of Smith[18] from 20 g (0.19 mol) of *m*-cresol. The product weighed 8.2 g (32%), bp 54–56 °C (5.0 mm) (lit.[19] bp 53 °C (3.0 mm)).

***m*-Nitrophenetole.** This compound was prepared according to the method of Smith[18] from 20 g (0.14 mol) of *m*-nitrophenol.[20] The product weighed 7.1 g (30%), mp 33–35 °C (lit.[21] mp 35 °C).

***p*-Iodoanisole.** This compound was prepared according to the method of Lucas and Kennedy[22] from 31 g (0.25 mol) of *p*-anisidine. The product weighed 20.6 g (34%), mp 50–51 °C (lit.[23] mp 49–52 °C).

**Typical Reaction Procedures.** All compounds were available commercially except for *m*-nitrophenetole and *p*-iodoanisole. The preparation of these compounds and the various iodonium salts is described above. For the reactions of the iodonium salts with sodium ethoxide, 50 mL of ethanol was reacted with the appropriate amount of sodium.

The sealed tubes were placed in an oil bath maintained at 71 ± 0.5 °C and allowed to remain there for 3 h. In every case, the sealed tube contained 0.0005 mol of the iodonium salt and 0.0007 mol of sodium ethoxide in 2 mL of ethanol solution. After completion of the reaction, the tube was opened, and the solution was neutralized with 85% phosphoric acid (usually requiring only 1 drop from a micropipet) and immediately analyzed on a Hewlett-Packard 5830 A flame-ionization gas chromatograph equipped with a 6-ft 10% SE-30 80–100 Chromosorb WAW column. Product identities were determined by comparison of retention times with those of known compounds and by "mixture VPC tests".

The reactions were carried out twice in batches of two at a time to assure maximum reproducibility. The yields of the reaction products were determined from the areas of the peaks which constitute part of the data output of the gas chromatograph. Three standard solutions, having compositions near the approximate value obtained from the reaction mixture, were then prepared for each component and subjected to VPC analysis; the approximate areas were obtained and plotted graphically vs. composition. The actual product compositions were then obtained directly from the graphs.

**Acknowledgment.** We thank Conicit (Venezuela) for support of this work.

**Registry No.** 5 (X = *m*-$CH_3$), 73377-23-8; 5 (X = *p*-$OCH_3$), 733-53-9; phenyl-*m*-tolyliodonium bromide, 73377-24-9; phenyl-*p*-methoxyiodonium bromide, 2665-61-4; *p*-tolyl-*m*-nitrophenyliodonium fluoroborate, 73377-26-1; sodium ethoxide, 141-52-6.

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