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Electronic Effects of Xenon-Containing Substituents

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Abstract

The electronic effects of xenon-containing substituents XeY, [Xe^+] and [XeF_2^+] in different solvents were calculated using the ^{19}F NMR method. The results obtained allow us to classify these substituents as the strongest σ-electron acceptors due to the strong positive inductive effect, while the resonance effect is small.

Keywords: inductive constant, resonance constant, arylxenon, NMR spectroscopy

INTRODUCTION

Correlation analysis is one of the most widely used methods to study the interaction of a substituent with the organic scaffold of a molecule, including the effect of the substituent on reaction directions. To determine the electronic constants of substituent X, the method proposed by Taft is widely used. This method is based on measuring the differences in chemical shifts in the ^{19}F NMR spectra (δ(F)) in the solutions of fluorobenzene and the isomers of 3-FC_6H_4X, 4-FC_6H_4X in a non-coordinating (cyclohexane, CCl_4) or weakly coordinating ($CHCl_3$, CH_2Cl_2) solvent, followed by the calculation of inductive (σ_I) and resonance (σ_R^0) constants according to equations [1, 2]:

$$\Delta\delta(F)_{meta} = \delta(F)(3\text{-}XC_6H_4F) - \delta(F)(C_6H_5F) = -7.10\sigma_I(X) + 0.60 \quad (1)$$

$$\Delta\delta(F)_{para} = \delta(F)(4\text{-}XC_6H_4F) - \delta(F)(C_6H_5F) = -29.5\sigma_R^0 - 7.10\sigma_I(X) + 0.60 \quad (2)$$

The electronic constants of several hundred substituents X of various types are presented in [3, 4], and most of those values have been determined using this method.

In 1989, the authors of [5] and [6] synthesised the first representatives of xenon-containing organic compounds: [C_6F_5Xe][$(C_6F_5)_nBF_{4-n}$] (n = 2, 3). Later on, other salts were obtained: [RXe][A] (R = aryl, polyfluoroalkenyl, polyfluorocycloalkenyl, alkynyl), [$C_6F_5XeF_2$][BF_4], compounds with two-coordinated xenon atom C_6F_5XeY, and their major structural, spectral and chemical properties were investigated [7–9].

In the present work, the electronic constants of xenon-containing substituents are assessed on the basis of experimental and literature data.

EXPERIMENTAL

Materials

Commercially available compounds were used: 3- and 4-FC_6H_4X (X = H, F, Cl, Br, I, C_6F_5, NH_2), C_6F_5X (X = H, Cl, Br, I, C_6H_5, C_6F_5, CN, CH_2CN, CCl_3, $CHCl_2$, COCl, COOH, $SiMe_3$, NH_2, OH, SH). The salts [3-$FC_6H_4N_2$][BF_4], [4-$FC_6H_4N_2$][BF_4], [$C_6F_5NH_3$][BF_4] [10], [C_6F_5Xe][AsF_6] [11] were obtained according to the published procedures. The solvents CD_3CN and EtCN were purified and

dried according to [12]. Acids HSO_3F, CF_3SO_3H were distilled before use.

Methods of investigation

The ^{19}F NMR spectra were recorded with Bruker WP 80 SY (Germany, 75.4 MHz) and Avance 300 (Germany, 282.40 MHz) instruments. Chemical shifts are given with respect to $CFCl_3$. NMR spectra were recorded using inliners (developed by H.-J. Frohn, Duisburg-Essen University, Germany), made of a copolymer of tetrafluoroethylene and hexafluoropropylene (FEP) (inner diameter 3.50 mm, outer diameter 4.10 mm, length 190 mm) and inserted into a standard NMR tube (outer diameter 5.00 mm) containing D_2O, CD_2Cl_2 or CD_3CN (0.1 mL). All procedures with arylxenon(II) compounds were carried out in the atmosphere of argon.

RESULTS AND DISCUSSION

Unlike for the derivatives of pentafluorophenylxenon(II) with the general formula $[C_6F_5Xe][(C_6F_5)_nBF_{4-n}]$ (n = 2, 3), the salts $[3\text{-}FC_6H_4Xe][BF_4]$ **1** and $[4\text{-}FC_6H_4Xe][BF_4]$ **2**, from the NMR ^{19}F spectra of which it is possible to determine the electronic effects of $[Xe]^+$ substituent, are thermally unstable above −50 °C [13]. They are insoluble in weakly coordinating dichloromethane, and their structure was confirmed relying on ^1H, ^{19}F, ^{129}Xe NMR spectra of solutions in the mixture of CD_3CN + EtCN (1 : 3 by volume) at −80 °C. This is a strongly coordinating basic solvent, and it was necessary to evaluate the possibility to use it for subsequent calculation of correlation constants according to Taft equations. For this purpose, we recorded NMR ^{19}F spectra of a series of compounds: 3- and 4-FC_6H_4X (X = H, F, Cl, Br, I, C_6F_5, NH_2, $[NH_3][BF_4]$) in the mixture CD_3CN + EtCN (1 : 3) at −80 and 24 °C, and confirmed the absence of noticeable influence of temperature on δ(F) values in comparison with their spectra in non-coordinating solvents, recorded at room temperature [1, 2]. The experimental values of inductive and resonance constants and the most reliable literature data [1–4] are presented in Table 1.

One can see that differences between both series are insignificant. This result allowed us to apply equations (1) and (2) to assess the electron effects of the positively charged xenon atom as a substituent. Taking into account solvent basicity, we may state that the calculated values $\sigma_I = 0.77$ and $\sigma_R^0 = 0.08$ correspond to the substituent $[Xe \cdot NCR]^+$ rather than to its non-solvated form $[Xe]^+$.

The salts of pentafluorophenylxenon(II) $[C_6F_5Xe][A]$ and the compounds C_6F_5XeY are substantially more stable than the salts of fluorophenylxenon **1** and **2** [7–9]. At a first glance, this opened the possibility to assess the electronic effects of xenon-containing substituents on the basis of ^{19}F NMR spectra by analogy with Taft's approach. However, it turned out that equations (1) and (2) are non-applicable here because of the mutual effect of five fluorine atoms. In 1972, the authors of [14] proposed alternative equations based on the analysis of ^{19}F NMR spectra of the solutions of compounds C_6F_5X (24 °C):

$$\delta(F)_{meta} = 5.21\sigma_I + 6.26\sigma_R^0 - 163.11 \quad (3)$$

$$\delta(F)_{para} = 8.91\sigma_I + 31.52\sigma_R^0 - 155.53 \quad (4)$$

TABLE 1

Effect of solvent and temperature on the electronic effects of substituents X in compounds FC_6H_4X according to ^{19}F NMR data

X	σ_I[a]	σ_I[b,c]		σ_R^0[a]	σ_R^0[b,c]	
	24 °C	24 °C	−80 °C	24 °C	24 °C	−80 °C
F	0.57	0.53	0.52	−0.33	−0.32	−0.32
Cl	0.43	0.42	0.41	−0.16	−0.17	−0.16
Br	0.49	0.46	0.45	−0.16	−0.16	−0.16
I	0.47	0.45	–	−0.12	−0.13	–
C_6F_5	0.25	0.22	–	0.03	0.03	–
NH_2	0.09	−0.01	–	−0.48	−0.53	–
$[NH_3][BF_4]$	0.60	0.61	–	−0.08	−0.07	–

Note. Here and in Tables 2–4: σ_I – inductive constant, σ_R^0 – resonance constant.

[a] According to data reported in [4].

[b] According to data from [1–3].

[c] Solvent CD_3CN + EtCN (1 : 3 by volume).

TABLE 2

^{19}F NMR spectra of compounds C_6F_5XeY and constants σ_I and σ_R^0

Y	Solvent	Temperature, °C	δ(F), ppm			σ_I	σ_R^0	References
			F-2,6	F-4	F-3,5			
F	CH_2Cl_2	−30	−128.8	−146.5	−156.1	1.45	−0.12	[15]
F	CD_2Cl_2	−78	−129.4	−146.9	−156.5	1.36	−0.11	[16]
F	CH_3CN	−30	−129.3	−148.4	−157.8	1.07	−0.08	[15]
F	RCN[a]	−58	−129.9	−148.6	−158.1	0.99	−0.06	[15]
Cl	CH_2Cl_2	−60	−130.8	−146.2	−155.5	1.61	−0.16	[17]
Cl	RCN[a]	−60	−131.0	−147.5	−157.0	1.25	−0.10	[17]
CN	CH_2Cl_2	−78	−131.5	−147.5	−156.4	1.42	−0.15	[16]
$OCOC_6F_5$	CD_2Cl_2	−10	−128.3	−144.7	−154.8	1.73	−0.15	[18]
C_6F_5	CD_2Cl_2	−78	−133.0	−154.2	−159.0	1.05	−0.25	[16]

[a] Solvent CD_3CN + EtCN (1 : 3 by volume).

Our verification reveals that the ^{19}F NMR spectra of C_6F_5X solutions, recorded in CD_3CN + EtCN (1 : 3 by volume) (X = H, Cl, Br, I, C_6H_5, C_6F_5, CN, CH_2CN, CCl_3, $CHCl_2$, COCl, COOH, $SiMe_3$, NH_2, OH, SH, $[NH_3][BF_4]$, $[N_2][BF_4]$) and in CD_2Cl_2 (X = H, Cl, Br, I, CN, CH_2CN, CCl_3, COCl, $SiMe_3$, NH_2, OH) at −40 °C exhibit almost no differences from those recorded in CCl_4 or CD_2Cl_2 at 24 °C ($\Delta\delta(F) \leq 0.5$ ppm), and equations (3), (4) can be used to calculate the constants σ_I and σ_R^0 in these solvents.

Results shown in Table 2 demonstrate the presence of strong positive inductive effect of XeY substituents and a weak resonance effect. In our opinion, these substituents are the strongest among the known σ-electron acceptors having no formally positive change. However, compound $C_6F_5XeOCOC_6F_5$ in the solid form is characterised by a very long Xe−O bond (2.367 Å) [18] and probably occupies an intermediate state between the derivatives of arylxenon(II) of the types of ArXeA and [ArXe][A]. Deshielding of fluorine atoms F-3,4,5 in the ^{19}F NMR spectra of C_6F_5XeY (Y = F, CN) passing from basic solvent CH_3CN or RCN to weakly coordinating dichloromethane corresponds to a decrease in the charge on xenon but not to ionisation of the Xe−Y bond. The latter has been confirmed by ^{129}Xe NMR spectroscopic data in both solvents, where spin-spin coupling constants ^1J(Xe, F) [16] and ^1J(Xe, C) were observed [17]. The smallest inductive effect manifests itself in compound $C_6F_5XeC_6F_5$, in which both Xe−C bonds are equivalent.

Below we present the values of σ_I and σ_R^0 constants of cations $[Xe^+]$, calculated according to equations (3), (4) from the ^{19}F NMR spectra of the solutions of $[C_6F_5Xe][A]$ in the solvents of different types (CD_3CN, water, SO_2 and acids).

The positions of ^{19}F NMR signals for $[C_6F_5Xe][A]$ in acetonitrole (CH_3CN) are weakly dependent on temperature and on the nature of anion [A] (Table 3). Consequently, the values of inductive constant σ_I are within a narrow range (1.6−1.8). An exclusion is compound $[C_6F_5Xe][HF_2]$ **3**, for which σ_I value occupies an intermediate position between those for C_6F_5XeF and the salt $[C_6F_5Xe][AsF_6]$ **4**, which depicts significant interaction of xenon with the fluorine atom of the anion.

The ^{19}F NMR spectrum of the aqueous solution of salt **4** (35 °C) [7] almost coincides with the spectrum in acetonitrile, and the constants in water are $\sigma_I = 1.63$, $\sigma_R^0 = -0.05$. In less basic SO_2 (0 °C) [7], the inductive effect of $[Xe^+]$ increases sharply ($\sigma_I = 1.98$), but the resonance effect remains negligibly small ($\sigma_R^0 = -0.06$). In strong acids (anhydrous HF, HSO_3F, CF_3SO_3H) this effect is extremely enhanced because the dissolution of $[C_6F_5Xe][A]$ salts proceeds due to anion salvation, while the cation becomes almost free. It follows from the data shown in Table 4 that σ_I values for all anions A are within the range 2.2−2.4.

The salts $[C_6F_5Xe][A]$ with low-nucleophilic anions A = BF_4, AsF_6 are insoluble in nonpolar weakly coordinating solvents, and it appears impossible to assess the electronic effects of $[Xe^+]$ in these media. Quite contrary, the salt $[C_6F_5Xe][B(CF_3)_4]$ is rather well soluble in CD_2Cl_2 and stable in solution at −80 °C, which allowed recording NMR spectra [20]. It turned out that under these conditions $\sigma_I = 2.45$ and $\sigma_R^0 = 0.10$. This means that the substituent $[Xe^+]$ is the strongest electron acceptor among the known ones, if its solvation by a solvent is minimal.

It could be expected that the substituent $[XeF_2^+]$ would exhibit higher electron acceptor

TABLE 3

^{19}F NMR spectra of compounds $[C_6F_5Xe][A]$ in CD_3CN and constants σ_I and σ_R^0

[A]	Temperature, °C	δ(F), ppm			σ_I	σ_R^0	Reference
		F-2,6	F-4	F-3,5			
$[HF_2]$	−40	−127.1	−144.6	−156.1	1.34	−0.03	[19]
$[B(OTeF_5)_4]$	24	−125.5	−141.8	−154.7	1.59	−0.01	[20]
$[SiF_5]$	24	−126.8	−143.0	−154.9	1.60	−0.06	[20]
$[AsF_6]$	24	−124.8	−141.3	−154.2	1.70	−0.03	[20]
$[AsF_6]$	−40	−125.1	−141.5	−154.3	1.69	0.03	[21]
$[BF_4]$	24	−124.8	−141.9	−154.7	1.59	−0.01	[20]
$[BF_4]$	−30	−125.5	−142.3	−155.0	1.53	−0.01	[22]
$[C_6F_5BF_3]$	−30	−125.1	−141.6	−154.3	1.69	−0.03	[6]
$[(C_6F_5)_2BF_2]$	−30	−125.2	−142.0	−154.7	1.60	−0.02	[11]
$[(C_6F_5)_3BF]$	20	−125.2	−142.0	−154.8	1.57	−0.02	[5]
$[B(C_6F_5)_4]$	24	−124.9	−141.1	−154.0	1.75	−0.04	[20]
$[B(CF_3)_4]$	24	−124.8	−141.0	−154.0	1.75	−0.03	[20]
$[B(CN)_4]$	24	−124.5	−140.9	−153.8	1.80	−0.05	[20]

TABLE 4

^{19}F NMR spectra of compounds $[C_6F_5Xe][A]$ in acids and constants σ_I and σ_R^0

[A]	Acid	Temperature, °C	δ(F), ppm			σ_I	σ_R^0	References
			F-2,6	F-4	F-3,5			
$[BF_4]$	HF	10	−123.2	−137.6	−151.4	2.30	−0.08	[a]
$[BF_4]$	HF	−10	−123.3	−137.9	−151.5	2.29	−0.09	[22]
$[BF_4]$	HF	−40	−123.6	−138.2	−151.8	2.22	−0.08	[20]
$[AsF_6]$	HF	−10	−123.2	−138.0	−151.6	2.33	−0.10	[a]
$[AsF_6]$	HF	−25	−122.9	−137.9	−151.6	2.26	−0.08	[a]
$[AsF_6]$	HSO_3F	24	−122.8	−137.6	−151.5	2.36	−0.10	[a]
$[AsF_6]$	HSO_3F	−10	−123.4	−138.1	−151.9	2.27	−0.09	[a]
$[AsF_6]$	HSO_3F	−40	−123.5	−138.5	−152.2	2.20	−0.08	[a]
$[AsF_6]$	CF_3SO_3H	24	−123.2	−137.7	−151.5	2.35	−0.10	[a]
$[AsF_6]$	CF_3SO_3H	−10	−123.3	−138.3	−151.9	2.25	−0.09	[a]
$[AsF_6]$	CF_3SO_3H	−40	−123.4	−138.7	−152.3	2.18	−0.08	[a]

[a] Present work.

ability in comparison with $[Xe^+]$. Compound $[C_6F_5XeF_2][BF_4]$ is the only representative of organic molecules containing xenon atom in oxidation degree 4+ [23, 24]. The signals from fluorine atoms of pentafluorophenyl group appear at δ(F) equal to −124.1 (F-2,6), −133.9 (F-4), −154.3 (F-3,5) ppm, (CD_3CN, −40 °C) and −123.4 (F-2,6), −128.5 (F-4), −150.1 (F-3,5) ppm (HF, −40 °C) [23]. Thus, inductive constants σ_I are equal to 1.25 and 2.16, while resonance constants σ_R^0 are equal to 0.33 and 0.25. Higher charge of xenon atom in $[C_6F_5XeF_2]^+$ (1.93) in comparison with $[C_6F_5Xe]^+$ (0.885) [24] is likely to promote more substantial solvation of $[XeF_2^+]$ in comparison with $[Xe^+]$ not only in CD_3CN but also in HF.

CONCLUSION

Comparison of constants σ_I and σ_R^0, calculated from ^{19}F NMR spectra of compounds $[3(4)\text{-}FC_6H_4Xe][BF_4]$, with the constants obtained for $[C_6F_5Xe][Y]$ reveals a substantial increase in the inductive effect of $[Xe^+]$ with an increase in the number of fluorine atoms in the aromatic ring with the conservation of the small resonance effect. The latter is explained by the small degree of conjugation of the π-system of aryl group with the corresponding orbitals of the large xenon atom, while the former is due to the joint electronic effect of five fluorine atoms. Coordination with electron-donor molecules (acetonitrile) or the formation of chemical bond Xe−Y causes a decrease in the induc-

tive effect of xenon-containing substituent. At the same time, it should be stressed that the calculated electronic constants based on the measurements of chemical shifts in the ^{19}F NMR spectra of compounds C_6F_5XeY, $[C_6F_5Xe][A]$, $[C_6F_5XeF_2][BF_4]$ are to be considered as approximate values because it is difficult to take into account the influence of several fluorine atoms on the positions of signals from indicator atoms F-3,5 and F-4, though in general they depict the electronic effects of xenon-containing substituents.

In spite of the fact that the methods of obtaining xenon-containing organic compounds and their major physicochemical properties have been described, the reactivity of these compounds is represented only scarcely. In particular, this relates also to the possible use of arylxenon(II) derivatives as organic catalysts. The theoretical pattern of intermolecular interaction of ArXeY and [ArXe][A] with some organic compounds has been presented in the recent publication [25] (see also the references therein related to the same area). However, in that case, the reaction centre is xenon atom, while the electronic constants characterise the effect of substituent on the reactivity of the organic group to which it is bound. These two respects together provide an objective characterisation of the reactivity of compounds under investigation, which will find application in planning and interpreting the experiments involving arylxenon derivatives.

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