Solvent Effects on Kinetics of the Reaction between 2-Chloro-3,5-dinitropyridine and Aniline in Aqueous and Alcoholic Solutions of [bmim]BF$_4$

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ABSTRACT: Rate constants, k_A, for the aromatic nucleophilic substitution reaction of 2-chloro-3,5-dinitropyridine with aniline were determined in different compositions of 1-(1-butyl)-3-methylimidazolium terafluoroborate ([bmim]BF$_4$) mixed with water, methanol, and ethanol at 25°C. The obtained rate constants of the reaction in pure solvents are in the following order: water > methanol > ethanol > [bmim]BF$_4$. In these solutions, rate constants of the reaction decrease with the mole fraction of the ionic liquid. Single-parameter correlations of log k_A versus normalized polarity parameter (E_T^N), hydrogen bond acceptor basicity (β), hydrogen bond donor acidity (α), and dipolarity/polarizability (π^*) do not give acceptable results in all solutions. Dual-parameter correlations of log k_A versus E_T^N and β also α and β gave reasonable results (e.g., in solutions of water with [bmim]BF$_4$, the correlation coefficients are 0.994 and 0.996, respectively). The proposed dual-parameter models demonstrate that the reaction rate constant increases with E_T^N, β, and α. The increase in the rate constant is attributed to hydrogen-bonding interactions (donor and acceptor) of the media with an activated complex of the reaction that has the zwitterionic character. © 2007 Wiley Periodicals, Inc. Int J Chem Kinet 39: 681–687, 2007

INTRODUCTION

The energetic level of molecules may be modified by interactions with surrounding species, and it may be difficult to relate chemical properties to molecular structures [1]. The solvent effects play a key role in many chemical and physical processes in solutions.

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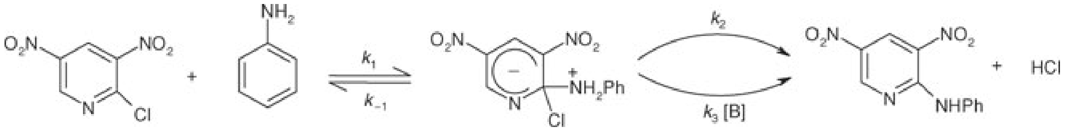


Scheme 1

The strong influence of solvents on chemical and physical processes (reaction rates, selectivity, chemical equilibria, position and intensity of spectral absorption bands and liquid chromatographic separations) has been well established [2]. Solvent effects are closely related to the nature and extent of solute–solvent interactions, locally developed in the immediate vicinity of the solutes. The study of solute–solvent interactions in binary mixed solvent systems is more complex than in pure solvents. The solute can be preferentially solvated by any of the solvents present in the mixture. On the other hand, solvent–solvent interactions can strongly affect solute–solvent interactions [1,3]. The problem in studying solvent effects on the reaction rates is to identify and assess the relative importance of various factors on the solvent effects.

Room temperature ionic liquids (RTILs) have recently attracted greater attention as environmentally benign alternatives to conventional organic solvents in a variety of synthetic [4,5], catalytic [6], and electrochemical applications [7], as a result of their unique physical and chemical properties and the relative ease with which these properties can be fine-tuned by altering the cationic or anionic moieties comprising the RTILs [5]. They continue to excite interest for a number of reasons. First, ionic liquids are environmentally friendly solvents, particularly because they have very low-vapor pressures under ambient conditions [8]. Second, they might provide improved reactivities in a number of chemical processes [9,10]. Finally, the fact that a wide range of cations and anions can be employed gives chemists the potential to design the solvents with specific properties. In such case, ionic liquids are described as "designer solvents" [8]. Among various ionic liquids used as reaction media, those based on dialkylimidazolium salts have attracted particular attention, as they possess a wide range of liquids and are easy to prepare and handle [10,11].

Several organic reactions were performed in ionic liquids successfully, but quantitative aspects of reactions in ionic liquids have been much less investigated [12,13]. The complexity of even the simple reactions in RTILs is significantly greater than those of reactions in molecular solvents. Therefore, there is a continuing challenge to understand the role of RTILs in the reactions [13,14].

In continuing our studies of solvent effects on organic reactions [15,16] and similar studies [17], we were interested in effects of mixtures on ionic liquids with molecular solvents. In this work, the solvent effects on the kinetics of nucleophilic heteroatomic substitution reaction of 2-chloro-3,5-dinitropyridine with aniline was studied in aqueous and alcoholic solutions of 1-(1-butyl)-3-methyl imidazolium tetrafluoroborate ([bmim]BF_4) at 25°C. In our previous studies, we reported the effects of various solvatochromic parameters on different organic reaction rates [16].

The suggested reaction mechanism of 2-chloro-3,5-dinitropyridine with aniline or similar primary or secondary amines is shown in Scheme 1 [16e,18].

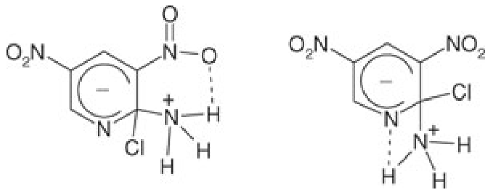
Application of the steady-state approximation gives Eq. (1) in which k_A is the observed second-order rate constant and B is a second molecule of the amine or an added base:

$$k_A = \frac{k_1\left(k_2 + k_3[\mathrm{B}]\right)}{k_{-1} + k_2 + k_3[\mathrm{B}]} \qquad (1)$$

Either the formation of an intermediate or its decomposition into products, in these reactions, may be considered as a rate-determining step. Therefore, different cases may be established, which have been reported elsewhere [16e]. If the formation of the intermediate is the rate-determining step, that is, (a) $k_{-1} \ll (k_2 + k_3\ [\mathrm{B}])$, then Eq. (1) reduces to $k_A = k_1$, and the amine does not catalyze the reaction. But, if the decomposition of the intermediate to products is the rate-determining step, that is, (b) $k_{-1} \gg (k_2 + k_3\ [\mathrm{B}])$, Eq. (1) is converted to Eq. (2) and the reaction is catalyzed by amines.

$$k_A = k_1 k_2 / k_{-1} + \left(k_1 k_3 / k_{-1}\right)[\mathrm{B}] \qquad (2)$$

The mechanism of both the catalyzed and the uncatalyzed reactions has been discussed in the presence of the primary and secondary amines [16e]. The reaction rate of this compound is much faster than that of 1-halo-2,4-dinitrobenzene, because of (a) electron-withdrawing of an aza group in addition to nitro groups and (b) the possibility of intramolecular hydrogen bonding between ammonium hydrogen and the aza group in the intermediate (Scheme 2). The latter is



Scheme 2

confirmed by high and negative activated entropy values of the reaction [18b].

EXPERIMENTAL SECTION

Materials

2-Chloro-3,5-dinitropyridine (m.p. 64°C) was obtained from Merck (Darmstadt, Germany) and purified by recrystallization from methanol-light petroleum as yellow needles. Aniline was purchased from Merck and purified by vacuum distillation. Methanol (>99.5%) and ethanol (>99.8%) were supplied by Merck and were used without further purification. Doubly distilled water was used in all solvent samples. 1-(1-Butyl)-3-methylimidazolium tetrafluoroborate (>98%), stored under argon, was purchased from Solvent-Innovation GmbH (Cologne, Germany) and was used as received. Karl Fischer titrations showed no detectable presence of water in freshly purchased [bmim]BF_4.

Kinetic Measurements

The kinetics of the reaction was studied spectrophotometrically, by running the reactions in the thermostated cells of spectrophotometer at 25°C. A GBC UV–vis cintra 40 spectrophotometer coupled with a thermocell was used with 1.00-cm silica cells, and the absorbance variation with time was recorded at $\lambda = 350$–360 nm (in different solvent compositions) for the product of reaction. The kinetics of reactions was studied under pseudo-first-order conditions with respect to 2-chloro-3,5-dinitropyridine. In all the cases, the infinity absorbance, A_∞, was determined experimentally for each kinetic run and used to calculate the reaction rate constant from Eq. (3):

$$\ln(A_\infty - A_t) = -k_{\mathrm{obs}}\, t + \ln(A_\infty - A_0) \qquad (3)$$

RESULTS AND DISCUSSION

To determine the rate-determining step of the reaction between 2-chloro-3,5-dinitropyridine and aniline, the influence of aniline concentration as a base on the reaction rate was studied at different compositions of solvents. The second-order rate constants of the reaction at various concentrations of aniline are shown in Table I.

Table I Second-Order Rate Constants at Different Concentrations of Aniline in Several Solvent Compositions at 25°C

	$10^2 \times k_A$ (M^{-1} s^{-1})			
	[Aniline] (M)			
x_1^a	0.0184	0.0369	0.0553	0.0922
Mixtures of [bmim]BF_4 and water				
0.0	122.1	120.3	121.5	121.0
0.3	9.76	9.72	9.63	9.65
0.6	3.88	3.76	3.70	3.80
1.0	1.60	1.72	1.70	1.63
Mixtures of [bmim]BF_4 and methanol				
0.0	34.2	33.4	32.5	33.6
0.3	6.59	6.56	6.63	6.62
0.7	2.39	2.33	2.30	2.28
Mixtures of [bmim]BF_4 and ethanol				
0.0	53.9	53.0	52.5	53.2
0.4	5.70	5.70	5.77	5.75
0.6	3.18	3.22	3.20	3.15

[a] x_1 is mole fraction of [bmim]BF_4.

These data show that no significant acceleration in the rate constant occurred with the increase in the aniline concentration. Similar results for this reaction in aqueous solutions of aliphatic alcohols have been observed [16e]. Similar results have also been reported for an aromatic nucleophilic substitution reaction of 1-fluro-2,4-dinitrobenzene and 1-chloro-2,4-dinitrobenzene with piperidine in aqueous solutions of various alcohols [16a,b,d]. Hence, the reaction is not catalyzed by a base. It can be concluded that the formation of the zwitterionic intermediate is the rate-determining step of the reaction, and the first case of Eq. (1) satisfies this situation.

The second-order rate constants of the reaction, k_A, in mixtures of [bmim]BF_4 with water, methanol, and ethanol were obtained at various mole fractions of [bmim]BF_4 at 25°C, and the results are summarized in Tables II–IV.

The solvatochromic parameters for solutions of [bmim]BF_4 with water, methanol, and ethanol at various compositions have been determined in our laboratory (Tables II–IV) [19]. Figure 1 demonstrates a plot of the reaction rate constant versus mole fraction of [bmim]BF_4. As can be seen, the rate constant of the reaction decreases sharply with the ionic liquid content. The second-order rate constant of the reaction follows

Table II Second-Order Rate Constants in Mixtures of Water with [bmim]BF$_4$ at 25°C

x_1	$10^2 \times k_A$ (M^{-1} s^{-1})	E_T^N	α	β	π^*
0.0	121.0	1.00	1.3	0.46	1.1
0.05	38.0	0.91	1.12	0.55	1.06
0.1	22.3	0.84	1.0	0.6	1.03
0.2	14.1	0.81	0.95	0.62	0.99
0.3	9.65	0.78	0.91	0.6	0.99
0.4	6.83	0.76	0.89	0.59	0.96
0.5	5.42	0.75	0.86	0.57	0.96
0.6	3.80	0.75	0.85	0.56	0.96
0.7	2.82	0.73	0.81	0.54	0.96
0.8	2.17	0.73	0.82	0.54	0.95
0.9	1.84	0.72	0.81	0.53	0.94
1.0	1.63	0.67	0.75	0.59	0.89

Table IV Second-Order Reaction Rate Constants in Mixtures of Ethanol with [bmim]BF$_4$ at 25°C

x_1	$10^2 \times k_A$ (M^{-1} s^{-1})	E_T^N	α	β	π^*
0.0	53.2	0.65	0.97	0.9	0.51
0.05	27.0	0.71	1.00	0.81	0.65
0.1	17.4	0.76	1.02	0.74	0.76
0.2	10.5	0.77	0.99	0.68	0.82
0.3	7.70	0.77	0.96	0.63	0.86
0.4	5.75	0.76	0.93	0.6	0.89
0.5	4.56	0.76	0.92	0.59	0.9
0.6	3.15	0.75	0.88	0.56	0.93
0.7	2.60	0.74	0.86	0.55	0.93
0.8	1.95	0.73	0.84	0.54	0.94
0.9	1.74	0.72	0.8	0.53	0.94
1.0	1.63	0.67	0.75	0.59	0.89

the following sequence: water > methanol > ethanol > [bmim]BF$_4$.

Although the normalized polarity parameter (E_T^N) of water is higher than that of ethanol and methanol, a single-parameter correlation of log k_A versus E_T^N does not give a reasonable result in all the solutions. For example in solutions of [bmim]BF$_4$ with methanol, the regression coefficient of log k_A versus E_T^N is 0.510. Also, there is no acceptable correlation between log k_A and hydrogen bond acceptor basicity (β) in any of the solutions, for example, it was observed in the solution of methanol with [bmim]BF$_4$ as in Eq. (4):

$$\log k_A = -4.202(\pm 0.386) + 4.791(\pm 0.626)\beta$$

$$(n = 12, r = 0.924, \mathrm{SE} = 0.181, F_{1,11} = 58.53) \quad (4)$$

where n, r, SE, and F are number of data, regression coefficient, standard error, and statistical Fisher number, respectively. Therefore, the normalized polarity parameter and hydrogen bond acceptor basicity of the media are not individually the main factor in determining solvent effects on the reaction rate. So, dual-parameter correlations of log k_A versus E_T^N and β were considered and are summarized in Eqs. (5)–(7). These parameters give good results in all the solutions:

In mixtures of water with [bmim]BF$_4$,

$$\log k_A = -8.123\ (\pm 0.445) + 6.776\ (\pm 0.261)\ E_T^N + 2.963\ (\pm 0.553)\beta$$

$$(n = 12, r = 0.994, \mathrm{SE} = 0.070, F_{2,10} = 364.12) \quad (5)$$

standardized coefficient of E_T^N is 1.075, and standardized coefficient of β is 0.221.

Table III Second-Order Rate Constants in Mixtures of Methanol with [bmim]BF$_4$ at 25°C

x_1	$10^2 \times k_A$ (M^{-1} s^{-1})	E_T^N	α	β	π^*
0.0	33.6	0.76	1.16	0.8	0.58
0.05	21.4	0.80	1.15	0.74	0.69
0.1	15.4	0.83	1.15	0.68	0.79
0.2	9.54	0.83	1.1	0.63	0.85
0.3	6.62	0.82	1.06	0.6	0.88
0.4	5.10	0.81	1.03	0.57	0.91
0.5	3.80	0.8	0.99	0.55	0.93
0.6	2.82	0.79	0.96	0.55	0.93
0.7	2.28	0.77	0.92	0.54	0.93
0.8	1.84	0.76	0.9	0.54	0.93
0.9	1.68	0.75	0.87	0.53	0.94
1.0	1.63	0.67	0.75	0.59	0.89

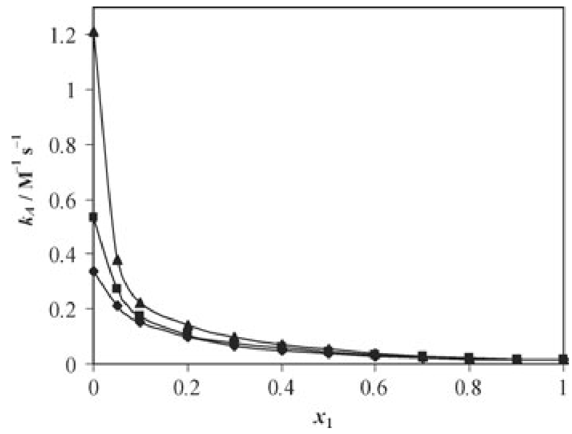


Figure 1 Second-order rate constants of reaction versus mole fraction of [bmim]BF$_4$ in its mixtures with methanol (♦), ethanol (■), and water (▲) at 25°C.

In mixtures of methanol with [bmim]BF$_4$,

$$\log k_A = -6.990\,(\pm 0.228) + 3.796\,(\pm 0.286)\,E_T^N + 4.492\,(\pm 0.147)\beta$$
$$(n = 12, r = 0.996,\ \mathrm{SE} = 0.042,\ F_{2,10} = 631.24) \quad (6)$$

standardized coefficient of E_T^N is 0.377, and standardized coefficient of β is 0.866.

In mixtures of ethanol with [bmim]BF$_4$,

$$\log k_A = -6.808\,(\pm 0.458) + 3.640\,(\pm 0.541)\,E_T^N + 4.544\,(\pm 0.180)\,\beta$$
$$(n = 12, r = 0.993,\ \mathrm{SE} = 0.064,\ F_{2,10} = 326.17) \quad (7)$$

standardized coefficient of E_T^N is 0.288, and standardized coefficient of β is 1.079.

The standardized coefficient or beta coefficient is the estimate of an analysis performed on variables that have been standardized, so that they have variance of 1. This is usually done to find out which of the independent variables have a greater effect on the dependent variable in the multiple regression analysis, when the variables are measured in different units of measurement. Before fitting the multiple regression equation, all variables (independent and dependent) can be standardized by subtracting the mean and dividing by the standard deviation. The standardized regression coefficients, then, represent the change in a dependent variable that result from a change in one standard deviation in an independent variable.

As can be seen, the second-order rate constant of the reaction increases with the increase in the normalized polarity parameter and hydrogen bond acceptor basicity. The activated complex leading to the zwitterionic intermediate (Scheme 2) is expected to have the zwitterionic character and to be favored by the increase in the normalized polarity parameter, because zwitterionic molecules were more stabilized in high-polar media than in the media with lower polarity. Also, hydrogen-bonding interactions of the media (solvent as acceptor with β parameter) with positive charge on the activated complex of the reaction stabilize the activated complex higher than the reactants; therefore, the increase in the β parameter accelerates the reaction rate. It is clear that in solutions of methanol and ethanol with [bmim]BF$_4$, the effects of β on the increase in the reaction rate is higher than that of E_T^N, because the standardized coefficient of β is higher than that of E_T^N.

The normalized polarity parameter is a blend of π^* (dipolarity/polarizability) and α (hydrogen bond donor acidity) of media. Thus, correlations of log k_A versus α in all the solutions were considered. The results demonstrate that the reaction rate increases with the increase in α. However, a good single parameter correlation between log k_A and α parameter was not observed in all the solutions. For example in solutions of methanol with [bmim]BF$_4$, the following result was obtained:

$$\log k_A = -4.580\,(\pm 0.394) + 3.290\,(\pm 0.390)\alpha$$
$$(n = 12, r = 0.936,\ \mathrm{SE} = 0.167,\ F_{1,11} = 71.13) \quad (8)$$

Hence, multiparameter correlations were considered. Multiparameter correlations of log k_A versus α and β demonstrate acceptable results in all the solutions (see Eqs. (9)–(11)):

In mixtures of water with [bmim]BF$_4$,

$$\log k_A = -6.686\,(\pm 0.344) + 4.043\,(\pm 0.133)\alpha + 3.263\,(\pm 0.478)\beta$$
$$(n = 12, r = 0.996,\ \mathrm{SE} = 0.060,\ F_{2,10} = 499.87) \quad (9)$$

standardized coefficient of α is 1.087, and standardized coefficient of β is 0.244.

In mixtures of methanol with [bmim]BF$_4$,

$$\log k_A = -4.862\,(\pm 0.090) + 1.960\,(\pm 0.129)\alpha + 2.649\,(\pm 0.190)\beta$$
$$(n = 12, r = 0.997, \mathrm{SE} = 0.037,\ F_{2,10} = 816.90) \quad (10)$$

standardized coefficient of α is 0.558, and standardized coefficient of β is 0.511.

In mixtures of ethanol with [bmim]BF$_4$,

$$\log k_A = -5.108\,(\pm 0.171) + 2.227\,(\pm 0.255)\alpha + 2.896\,(\pm 0.185)\beta$$
$$(n = 12, r = 0.996,\ \mathrm{SE} = 0.051,\ F_{2,10} = 514.41) \quad (11)$$

standardized coefficient of α is 0.383, and standardized coefficient of β is 0.688.

To show the efficiency of the suggested dual-parameter correlations, for example, the calculated values of the reaction rate constants in mixtures of methanol with [bmim]BF$_4$ using Eq. (10) have been plotted vs. the experimental values. Figure 2 shows a good agreement between the experimental and the calculated values of log k_A. Thus, unlike what has been reported in a few papers [13], results indicate that the media effects on this reaction can be described by means of the solvatochromic parameters in the presence of the ionic liquid in conventional solvents.

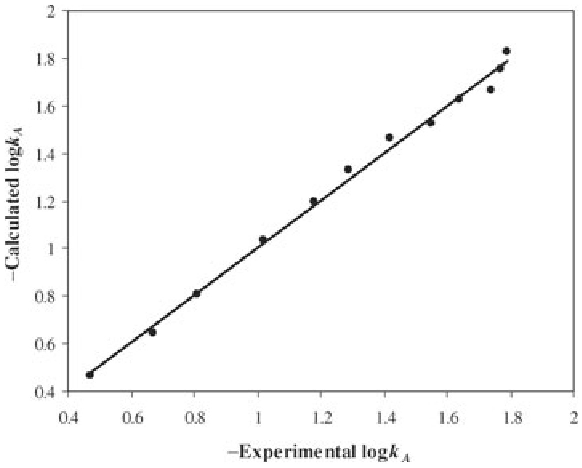


Figure 2 Plot of the calculated values of $-\log k_A$ by Eq. (10) versus the experimental values of it in mixtures of methanol with [bmim]BF_4 at 25°.

It is clear that the reaction rate constant increases with α and β in all the solutions. The negative charge of the activated complex of the reaction is on a pyridine ring. Hydrogen-bonding interactions of the media with negative charge on the activated complex of the reaction stabilize the activated complex more than the reactants; therefore, the reaction rate increases with the α parameter. The activated complex leading to the intermediate of the reaction has the zwitterionic character with positive charge on nitrogen of aniline and negative charge on the pyridine ring. Hydrogen-bonding interactions of media (donor and acceptor) with the charges on the activated complex stabilize the activated complex more than the reactant of the reaction. Thus, the reaction rate constant increases with hydrogen bond donor and acceptor parameters. As can be seen, in solutions of water with [bmim]BF_4 and methanol with [bmim]BF_4, the effect of α on the increase in the reaction rate is higher than β, because in these solutions the standardized coefficient of α is higher than that of β.

The α parameter of RTILs is largely affected by the nature of the cation, but there is also a smaller anion effect [20]. It has been known that in [bmim]BF_4 all three imidazolium ring hydrogen atoms are acidic. The α value for [bmim]BF_4 is moderately high but is lower than that of water, methanol, and ethanol [19]. The β parameter of RTILs is mainly dominated by the nature of the anion. The anion of [bmim]BF4 is known to have a compact structure, possessing much weaker basicity in comparison to alcohols. Then, [bmim]BF_4 has lower hydrogen bond donor acidity and hydrogen bond acceptor basicity relative to water (except for β), methanol, and ethanol. Therefore, the second-order rate constant of the reaction is in the following sequence: water > methanol > ethanol > [bmim]BF_4.

CONCLUSIONS

Changes in the solvent composition showed a dramatic effect on the rate of the aromatic nucleophilic substitution reaction of 2-chloro-3,5-dinitropyridine with aniline. The second-order rate constants of the reaction represent a falloff with the mole fraction of [bmim]BF_4. Unlike many reactions, the rate of the reaction decreases with the addition of [bmim]BF_4. In all these cases, formation of the zwitterionic intermediate is the rate-determining step of the reaction. Normalized polarity, hydrogen bond donor acidity, and hydrogen bond acceptor basicity of media have parallel and positive effects on the rate of the reaction. Thus, solvatochromic parameters of media can describe solvent effects on the reaction rate and represent a theoretical model for similar cases in the ionic liquid mixed with molecular solvents. The results of dual-parameter correlations of log k_A versus E_T^N and β (also α and β) in all the solutions represent improvements with regard to the single-parameter models.

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