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DISCUSSION

On the "Negative Effect of Nucleophilic Solvation" in Monomolecular Heterolysis

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Abstract—Critical analysis of the evidence for the operation of the "negative effect of nucleophilic solvation" in the monomolecular heterolysis reactions of organic compounds was performed. The existence of this effect is not proved.

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The "negative effect of nucleophilic solvation" was first described by Dvorko and co-workers in [1, 2]. This effect consists in a decrease of the rate of monomolecular heterolysis reactions of some substrates with the increasing solvent nucleophilicity. In the following time the authors obtained a large amount of experimental data that in their opinion confirmed the existence of this effect [3–6]. In turn the manifestation of this effect, according to the authors' idea, is an evidence for an additional intermediate in the course of monomolecular heterolysis, the "spatially" separated ion pair [7]. We believe that these conclusions require a critical analysis.

According to the explanation given in [3, 5, 6], the nucleophilic solvation of the contact ion pair stabilizes this intermediate and impede its further separation. The transfer of the nucleofuge to a nearby cavity between the solvent molecules occurs in the nonequilibrium solvation regime. In case of bipolar solvents this process requires an additional energy for the destruction of chelate bonds formed by the solvent molecules with the ions of the contact ion pair. This energy expenditure is not compensated in the transition state by the resolvation of the ions, since the solvation shell rearrangement lags behind the nucleofuge movement.

The "negative effect of nucleophilic solvation" is most pronounced in protic solvents forming strong hydrogen-bonded clusters with contact ion pairs, and with the substrates whose reaction site is rearside-shielded and therefore the solvent nucleophilic assistance at the ionization stage is precluded. According to the authors' view, the nucleophilic solvation reduces the rate of the heterolysis of adamantyl derivatives in protic solvents by 2–3 orders of magnitude compared to aprotic solvents. Since this effect is operative also in aprotic solvents, its true value in protic environments should be at least 3–4 orders of magnitude.

Whether at all the *specific nucleophilic* solvation of the adamantyl carbocation *from the front* in the contact ion pair can be strong enough is questionable. If the solvation in question is not strong enough due to steric reasons, it can not significantly affect the energy of the solvation/desolvation of the moving nucleofuge since no stable chelate stucture is formed. The isolation of this effect against the background of the dispersion and Coulomb interactions seems completely unrealistic. The molecule(s) of a nucleophilic aprotic solvent may displace the nucleofuge and block further reactions of the carbocation, but such a particle is no longer a contact ion pair.

It was found in [8, 9] that after the dissolution of 2-bromo-2-methylpentane in dimethylformamide a substance is quickly formed (ca. 20% of the initial substrate concentration), which under the action of water quickly liberates the bromide ion whilst on dilution with carbon tetrachloride its concentration in the solution decreases gradually. It was suggested that it may be a relatively inert (in the absence of proton-donor additives) complex of the contact ion pair or covalent substrate with the solvent. Dvorko and co-workers believe that this data indicate the possibility of slowing the separation of the contact ion pair by nucleophilic solvation. But if even in DMF the contact ion pair with the solvent formed only about 20% of the inactive complex, in less nucleophilic aprotic solvents (the vast majority of solvents) the amount of the formed inactive complex will be even smaller, and the effect of such “dead-end equilibrium” will not appreciably affect the reaction rate. In protic solvents such intermediates should be located on the reaction coordinate of solvolysis, and the negative effect of the solvent nucleophilicity on the rate of ions separation, in principle, cannot be excluded. The authors’ reference to the publication [10], which supposedly points to the need of destruction of hydrogen bonds in the solvation shell of the contact ion pair during its transformation into the solvent-separated ion pair, is not proper. In fact, in the mentioned paper the *ionization* of the substrate (that is, the formation of the contact ion pair) is considered.

We think that general considerations cannot provide a well-grounded conclusion about the existence and strength of the “negative effect of nucleophilic salvation.” So let us turn to the experimental data.

There exist several scales of the solvent basicity/nucleophilicity, described, for example, in [11]. The authors of the considered hypothesis agree that the Grunwald–Winstein equation $\log (k/k_0) = mY + lN$ is of little use for the assessment of the effect of solvent nucleophilicity [12]. However, in the same paper they use the value of coefficient l taken from the literature as an evidence of the “negative effect of nucleophilic salvation.” The assessment of the comparative suitability of different scales of solvent parameters for the correlation of solvation effects is beyond the scope of this paper. Therefore, in the following calculations we will use only the Koppel–Palm equation (1), which the authors of the hypothesis used most often in their calculations.

$$\log k = a_0 + a_1 f(\varepsilon) + a_2 f(n) + a_3 B + a_4 E + a_5 \delta^2. \qquad (1)$$

We will not consider the compensation effect (i.e. the activation enthalpy decrease with increasing and simultaneous the negative value of the activation entropy in the series of solvents [13, 14]) as the “negative effect of nucleophilic salvation.” In our opinion, such a general phenomenon *a priori* cannot be an argument in favor of any fine detail of a reaction mechanism. From the most general considerations, the more effective solvation that reduces the activation enthalpy will lead to the formation of more ordered solvation shell, that is, to more negative activation entropy. Thus, we will consider only the influence of the solvation on the reaction rates.

Before we turn to the analysis of the relevant experimental data, it is mandatory to make some remarks on the general requirements to the statistical analysis of experimental data. These requirements are obvious, but, unfortunately, they are often ignored by the researchers, which not infrequently leads to misunderstandings.

(i) Any calculated values do not contain any information until their precision is considered, e.g. the confidence intervals at a given confidence level are calculated. For example, it is of no use to discuss the meaning of “pure” coefficients a_i in Eq. (1), even if the multiple correlation coefficient is close to unity. To get a good enough multiple correlation it may be sufficient to have good correlation of the dependent variable with any of independent parameters when other parameters are statistically insignificant, despite their non-zero values. Unfortunately, the authors of the hypothesis in their papers only rarely give the values of the confidence intervals.

(ii) Data sets with different statistical parameters should not be combined into the aggregate array. For example, if we merge two sets of solvents in *only one* of which some effect occurs, we will “see” that effect in the aggregate set of solvents (though perhaps with poorer correlation parameters). In the present case we are talking about the sets of protic and aprotic solvents, the difference between which is constantly emphasized by Dvorko et al. [4, 6].

(iii) While analyzing the statistical significance of a factor in the multiparameter regressions, the possibility of the multicollinearity effect should be borne in mind. Multicollinearity is the presence of relationships between “independent” parameters X_i and X_j described by the pair correlation coefficients r_{ij}. More generally, multicollinearity occurs when a linear combination of independent parameters correlates with a parameter that is not a member of that set. This phenomenon can lead to a distortion of the regression coefficients for the corresponding parameters, and even change their signs [15]. Nonorthogonality of the argument scales leads to the effect of “swapping” of the respective coefficients a_i and a_j [16] and increases the standard deviations of the regression coefficients [15, 17]. The upper limit of r_{ij} when the results of multiparameter regression still deserve attention, apparently, is not yet defined [16]. In [17] the pair correlation coefficient $r_{ij} = 0.824$ is considered as indicating a strong correlation of the “independent” parameters. On the other hand, some researchers suggest that a multicollinearity is harmful only when $r_{ij} \geq R$, where R is the coefficient of multiple correlation regression [18].

Thus, special care is needed in data handling and interpreting when the multicollinearity of independent parameters is present. It is necessary to check different combinations of independent parameters, but a small difference in the multiple correlation coefficient cannot be taken as a convincing evidence in favor of a particular model compared to another one.

(iv) Finally, the calculated regression coefficients should be realistic. For example, the meaning of a_0 parameter in the Koppel–Palm equation is log k of a given reaction in the gas phase or, more precisely, in a “non-solvating solvent,” regardless of the use of full or partial set of independent parameters. For the reactions of unimolecular heterolysis its magnitude is unlikely to be higher (and more likely to be lower) than the value of log k in nonspecifically solvating low polarity solvents such as cyclohexane and, more so, benzene.

With this in mind let us analyze the data presented by Dvorko et al. The rate constants and solvent parameters are taken from [12]. In all cases, we will consider the results of our calculations, since the authors of the original papers used different scales of the solvent parameters in Eq. (1). Confidence intervals were calculated at 95% confidence level.

First of all, does the *in quotes* negative effect of the nucleophilic solvation exist in aprotic solvents? In a recent review [6] Dvorko et al. cite only two examples where this effect allegedly manifested itself in a range of aprotic solvents: the reactions of heterolysis of 2-bromo-2-methyladamantane [19] and *p*-methoxyneophyl tosylate [20]. In the first case, after the exclusion of the points for the *sym*-tetrachloroethane (deviation of the calculated value from the experimental value $\Delta\log k = 1.62$) we obtained Eq. (2):

$$\log k = (-13.5\pm2.5) + (10.3\pm3.4)f(\varepsilon) + (6.8\pm9.5)f(n) - (0.02\pm0.32)B + (0.12\pm0.06)E + (0.17\pm0.28)\delta^2, \quad n\ 26,\ R\ 0.951. \tag{2}$$

If, as the authors did, we exclude also the points of methylene chloride, chloroform, and 1,2-dichloroethane, a “good five-parameter dependence” [Eq. (3)] is obtained:

$$\log k = (-13.5\pm2.0) + (8.6\pm2.5)f(\varepsilon) + (5.0\pm7.6)f(n) + (0.22\pm0.25)B + (0.06\pm0.06)E + (0.41\pm0.24)\delta^2, \quad n\ 23,\ R\ 0.979. \tag{3}$$

Obviously, in this case we cannot talk about any “negative effect of nucleophilic salvation.” It is interesting that the authors of the original article also agreed with this statement: “In aprotic solvents, the rate of bromide (**II**) heterolysis does not depend on the nucleophilic properties of the medium” [19].

In the last case, the authors found no effect of the aprotic solvent nucleophilicity on the reaction rate using the Kamlet–Taft equation. The results of the calculations according to Eq. (1) have not been included in the article, so there is a reason to suspect that its use also did not allow to find the desired effect. Finally, the authors took one parameter of the Kamlet–Taft equation and combined it with the nucleophilicity parameter of Eq. (1). The degree of conclusiveness of such manipulation remains unclear.

In the above mentioned review there are three references to the manifestation of the “negative effect of nucleophilic solvation” in the sets of dipolar aprotic solvents [21–23]. In the article [23] were studied the activation parameters, and in two other papers there was no mention about the observation of this effect in dipolar aprotic solvents. We therefore continue our analysis of the data [19], the more so that the studied there 2-bromo-2-methyladamantane, according to the authors’ words, is highly sensitive to specific solvation. For all dipolar aprotic solvents listed in the article we obtained Eq. (4).

$$(\log k = (-16\pm69) + (20\pm104)f(\varepsilon) - (1\pm9)f(n) - (0.1\pm1.4)B + (0.02\pm0.14)E + (0.4\pm1.1)\delta^2, \quad n\ 10,\ R\ 0.973. \tag{4}$$

The maximum deviation is only 0.34, and there are no statistical reasons to exclude any point. When only the parameters B and δ^2 are used, the coefficient at the parameter B remains insignificant (–0.08±0.5). Thus, even for this substrate in the subset of dipolar aprotic solvents the effect in question is not observed.

As for the protic solvents, the analysis of all data obtained by the authors would occupy too much space. Our spot-check indicates that in some cases the possibility of manifestation of this effect cannot be excluded, but its existence is also difficult to prove, particularly because of the multicollinearity of parameters within the set of alcohols. To reduce the multicollinearity, the data for other classes of protic solvents should be included, but in most cases such data for a sufficient number of solvents is either absent [22, 24], or their inclusion leads to much worse correlations [19, 25].

For example, using the full equation (1), for the set of nine alcohols presented in [19] R = 0.972, and with the inclusion of acetic acid, R = 0.926. It is clear that the exclusion of any set of parameters cannot make the latter correlation satisfactory. And even with the satisfactory correlation coefficient for the set of alcohols we can not draw any conclusions from the caclulation results [Eq. (5)]:

$$\log k = (4\pm32) - (6.3\pm24)f(\varepsilon) + (2\pm21)f(n) \\ - (3\pm8)B + (0.00\pm0.13)E + (0.5\pm1.1)\delta^2. \qquad (5)$$

Apparently, the problem here lies in the parameter multicollinearity (r_{BE} = 0.78) and a very small number of degrees of freedom for such a large number of independent parameters. When using only the parameters of the basicity and cohesion, the effect of the “negative nucleophilic solvation” seems to manifest itself, and there are no reasons to exclude any point ($\Delta\log k$ = 0.12–0.35) [Eq. (6)].

$$\log k = (3\pm7) - (3.6\pm2.3)B + (0.35\pm30)\delta^2, \qquad (6) \\ n\ 9,\ R\ 0.961.$$

When replacing the basicity parameter with the parameter of electrophilicity, the correlation deteriorates (R = 0.872), but after excluding one clearly deviating point (for benzyl alcohol $\Delta\log k$ = 1.19, the average deviation after its exclusion 0.12), the correlation is even better than when using the basicity parameter [Eq. (7)].

$$\log k = -(8.2\pm1.1) - (0.01\pm0.03)E + (0.51\pm0.37)\delta^2, \qquad (7) \\ n\ 8,\ R\ 0.984.$$

The insignificance of the solvent electrophilicity parameter in the resulting equation may seem surprising, but to reveal the cause of this phenomenon it is enough to look at the correlation coefficient between “independent” parameters: $r_{E\delta^2}$ = 0.937.

It is clear from the foregoing how inaccurate and unreliable for our purposes are the models used, and therefore how cautiously one should interpret the obtained data. It should be emphasized that the equations of type (6) *a priori* are doubtful as they by default consider insignificant the effect of solvent electrophilicity. Such an approach is either incorrect, or pretend to be a new theory in which the electrophilicity of environment does not affect the rate of S_N1 reactions.

As another example, let us consider the reaction of heterolysis of *tert*-butyl chloride [2], where Dvorko et al. also found the manifestation of the “negative effect of nucleophilic solvation” in a set of protic solvents. This is a classical reaction, and there is the largest number of experimental data available concerning it. The set of 14 protic solvents includes alcohols, water, and two carboxylic acids (acetic and formic) [Eq. (8)].

$$\log k = (-14\pm7) - (23\pm15)f(\varepsilon) + (6\pm12)f(n) \\ - (1.9\pm1.4)B + (0.003\pm0.05)E + (0.18\pm0.13)\delta^2, \qquad (8) \\ n\ 17,\ R\ 0.972.$$

The parameter of the solvent electrophilicity is insugnificant, which seems illogical. There is no one distinctly deviating point there, but if we exclude both carboxylic acids, the correlation improves dramatically [Eq. (9)].

$$\log k = (-7.4\pm4.4) - (2.7\pm12.3)f(\varepsilon) + (4.8\pm6.0)f(n) \\ - (1.05\pm0.70)B + (0.05\pm0.04)E + (0.15\pm0.09)\delta^2, \qquad (9) \\ n\ 15,\ R\ 0.994.$$

After excluding the insignificant polarity and polarizability parameters we obtained Eq. (10).

$$\log k = (-6.6\pm2.0) - (1.20\pm0.54)B + (0.039\pm0.024)E \\ + (0.17\pm0.06)\delta^2, \qquad (10) \\ R\ 0.991.$$

Although formally Eq. (10) meets all statistical criteria, the value of a_0 is beyond the reasonable range: the value of log k in *tert*-pentyl alcohol is –8.8, and in heptane –15.8. If the parameters B and E (r_{BE} = 0.81) are used separately, the confidence intervals again are much narrower though the correlaton coefficients are lower [Eqs. (11), (12)].

$$\log k = (-11.1\pm0.8) + (0.087\pm0.010)E + (0.07\pm0.04)\delta^2, \qquad (11) \\ R\ 0.973;$$

$$\log k = (-3.32\pm0.9) - (2.0\pm0.3)B + (0.26\pm0.05)\delta^2, \qquad (12) \\ R\ 0.982.$$

Solvation characteristics of *tert*-butyl chloride in protic solvents are similar to those of 2-methyl-2-bromoadamantane, but the quality of correlations for the *tert*-butyl substrate is much higher. However, in our opinion, in this case the existence of the “negative effect of nucleophilic solvation” in a set of protic solvents cannot be unambiguously accepted.

In another attempt to prove the operation of the “negative effect of nucleophilic solvation” Dvorko et al. chose combined sets of protic and dipolar aprotic solvents that allegedly gave particularly good correlations [19, 25]. The authors considered it possible to create such sets solely on the basis of their arguments that in dipolar aprotic solvents this effect should also occur. We have already mentioned the dangers of such data manipulation, but let us follow the authors [19].

In a set of 7 protic and 7 dipolar aprotic solvents[1], Dvorko et al. refer to three-parametric correlation (13).

$$\log k = (-16\pm4) - (29\pm9)f(\varepsilon) - (1.3\pm0.5)B + (0.034\pm0.010)E, \quad R\ 0.957. \tag{13}$$

The exception of the B parameter actually destroys the correlation ($R = 0.760$), and the authors believe that this fact is a sufficient proof of their point of view. But let us begin with a more general equation, including the cohesion parameter [Eq. (14)].

$$\log k = (-15\pm2.5) - (21\pm6)f(\varepsilon) - (0.7\pm0.4)B + (0.015\pm0.010)E + (0.31\pm0.15)\delta^2, \quad R\ 0.988. \tag{14}$$

In this case, the exclusion of any of the specific salvation parameters only slightly impairs the correlation:

$$\log k = (-15\pm4) - (15\pm9)f(\varepsilon) + (0.004\pm0.013)E + (0.51\pm0.16)\delta^2, \quad R\ 0.965;$$

$$\log k = (-13\pm3) - (14\pm6)f(\varepsilon) - (0.36\pm0.42)B + (0.49\pm0.11)\delta^2, \quad R\ 0.974.$$

From the viewpoint of statistics, it is not clear why the authors chose to exclude namely the cohesion parameter, although this leads to a worse correlation [Eq. (13)]. The considered data can be "satisfactorily" correlated even excluding both the basicity and electrophilicity parameters [Eq. (15)].

$$\log k = (-14\pm3) - (13\pm7)f(\varepsilon) + (0.54\pm0.11)\delta^2, \quad R\ 0.964. \tag{15}$$

Thus, in our calculations the "negative effect of nucleophilic solvation" was *formally* statistically confirmed only in protic solvents and in the specially selected set of protic and aprotic solvents. However, the values of some regression coefficients in protic solvents were unrealistic, and the presence of multicollinearity probably did not allow to estimate the statistical significance of the parameters. As for the combined sets of protic and aprotic solvents, there is a wide opportunity for the selection of solvents to fit a pre-created model. For example, in [19, 25] highly basic dipolar aprotic solvents like carboxylic acids amides and dimethyl sulfoxide were not studied for some reason.

Worth noting are the differences found by Dvorko et al. between the solvation effects in the heterolysis of different substrates [12]. For some compounds (benzhydryl bromide, *tert*-butyl chloride), common linear dependences of log k vs the Dimroth E_T parameter obey in all solvents, while in other cases in protic and aprotic solvents are observed the linear segments that differ by their slope and/or height. For example, the rate constant of the heterolysis of 2-bromo-2-methyladamantane in protic solvents is lower by 2–3 orders of magnitude than it would be in aprotic solvents with the same value of E_T. The cause of this difference, according to Dvorko et al., is the "negative effect of nucleophilic solvation." In our opinion, the diversity of the observed dependences cannot be attributed with certainty to the manifestation of this effect. For example, in all aprotic solvents the dependences log k – E_T for all substrates are linear, which does not permit to distinguish the dipolar aprotic solvents as a separate class where the effect is operative, on the contrary to the other aprotic solvents. On the other hand, observations of Dvorko et al. once again show the difference between protic and aprotic solvents, which does not permit including these solvents in common sets for the correlation analysis.

In our opinion, the presence of the "negative effect of nucleophilic solvation" in protic solvents is not currently proved, but it cannot be completely excluded. Serious arguments in favor of this effect in aprotic media are virtually absent.

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[1] Cyclohexane is excluded, for the authors included it erroneously as the eighth dipolar solvent. In this set of solvents also there is some multicollinearity between independent variables: $r_{E\delta^2}$ = 0.675, $r_{f(\varepsilon),E}$ = 0.492.

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