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Solvatochromism of Heteroaromatic Compounds: VI.[1] Comparison of the Empirical and Theoretical Approaches to Description of Solvatochromism in Nonspecific Solvation of Nitropyrazoles

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Abstract—The effect of nonspecific solvation on the long-wave absorption band in the UV spectra of isomeric nitropyrazoles was studied. The aprotic inert and aprotic protophilic solvents exert different spectroscopic effects. In the former solvents, the solvatochromism is mainly governed by orientation interactions, and in the latter solvents, along with the orientation interactions, the UV spectrum is appreciably influenced by the induction, polarization, and dispersion interactions, which is due to the presence of readily polarizable nonbonding electron pairs and/or a π-electron systen in molecules of such solvents.

The principal origin of solvatochromism was elucidated long ago. However, the views on its specific features are still very approximate, and the existing theories are insufficiently complete to describe with fair accuracy the actual solvent effects. Therefore, empirical approaches are widely used. In particular, relatively recently Kamlet, Taft, *et al.* developed a method of solvatochromic comparison. Using this method, they obtained a set of empirical parameters of solvents taking into account both specific and nonspecific interactions [2, 3]. These parameters were successfully used to describe regular trends in many chemical phenomena [3]. In our previous studies [1, 4–7], we used these parameters when studying the effect of solvation on the UV spectra of neutral molecules and anions of a number of nitrogen-containing five-membered heterocycles. These studies furnished interesting information on the rotation isomerism of 1-ethyl-2-formylimidazole [1], tautomerism of 3(5)-nitropyrazole [6], and structure of solvation complexes of NH-containing pyrazoles and pyrroles [4–7]. The fruitfulness of using the Kamlet–Taft solvatochromism parameters calls for detailed elucidation of their physical sense.

In terms of the Kamlet–Taft approach, in the absence of specific solvation the position of the UV absorption maximum (ν_{max}) of a compound depends only on the polarity parameter π^* of the medium [8]:

$$\nu_{max} = \nu_0 + c\pi^*. \qquad (1)$$

In this equation, ν_0 is a constant characterizing the spectrum of a molecule in a fully inert solvent ($\pi^* = 0$); $c = A\pi^*_m$, where π^*_m is the polarity parameter of the compound under consideration [3]. Introduction of this parameter into the solvatochromism equation is based on the general knowledge; it corresponds to the ability of a solvent to enter electrostatic and/or electrodynamic interactions with the solvation shell molecules. Such interactions can be described theoretically using the functions of universal interactions (see, e.g., [9]):

$$\nu_{max} = \nu_{gas} + c_1 f_1 + p f_2, \qquad (2)$$

where $f_1 = [(2n^2 + 1)/(n^2 + 2)]\{[(\varepsilon - 1)/(\varepsilon + 2)] - [(n^2 - 1)/(n^2 + 2)]\}$ and $f_2 = (n^2 - 1)/(n^2 + 2)$. The parameters of this equation have a clear physical sense: $\nu_{gas} = \nu_{max}$ in the gas phase, $c_1 = 2\mu_g(\mu_g - \mu_e \cos\varphi)/a^3$, where $\mu_{g,e}$ is the dipole moment in the ground (g) and excited (e) states; φ is the angle between the vectors of the moments; a is the Onsager radius; the parameter p is related to the induction, dispersion, and polarization interactions [9]. Equation (2) includes the function of the dipole moment of the solute in the ground state as the factor influencing its spectroscopic features in a wide range of solvents of different nature. In this work, using isomeric nitropyrazoles **I**–**V** as example, we studied empirically the correla-

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[1] For communication V, see [1].

Table 1. Dipole moments of isomeric nitropyrazoles in various media

Comp. no.	Aprotic inert media, μ^{25}, D		Aprotic protophilic media, μ^{25}, D			Free molecule, μ(6–31G*), D
	hexane	CCl_4	xylene	benzene	dioxane	
I	3.71	3.64	3.64	3.63	3.60	4.78
II	[a]	5.75	[a]	6.26[b]	6.20[b]	7.26
III	[a]	4.35	4.62	4.61	4.83	5.69
IV	4.65	4.53	4.62	4.63[b]	4.89[b]	5.47
V	4.16	4.07		4.04	4.19	4.77

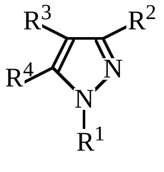
[a] The attainable concentration is insufficient for measuring the dipole moment. [b] Data of T.N. Aksamentova, I.G. Krivoruchek, V.N. Elokhina, A.I. Vokin, V.A. Lopyrev, and V.K. Turchaninov will be published.

Table 2. Solvatochromism of the long-wave absorption band in the UV spectra of isomeric nitropyrazoles **I**–**V** in aprotic inert solvents

Solvent	Solvent parameters[a]			ν_{max}, cm^{-1}				
	π^*	f_1	f_2	**I**	**II**	**III**	**IV**	**V**
Hexane	–0.08	0.00	0.23	38 200	39 700	37 800	36 500	34 650
Pentane	–0.08	0.00	0.22	38 250	39 700	37 800	36 400	34 700
Heptane	–0.02	0.00	0.24	38 150	39 500	37 700	36 400	34 550
Cyclohexane	0.00	0.00	0.26	38 100	39 550	37 650	36 200	34 500
Octane	0.01	0.01	0.24	38 100	39 600	37 700	36 300	34 600
Decane	0.03	0.00	0.25	38 100		37 650	36 300	34 500
Tetrachloromethane	0.28	0.02	0.28	37 900	38 800	37 100	35 900	34 000
1-Chlorobutane	0.39			37 700	38 350	36 850	35 600	33 900
Chloroform	0.58	0.37	0.27	37 500		36 500	35 300	33 500
Pentachloroethane	0.62	0.25	0.30	37 500	37 800	36 550	35 300	33 500
1,2-Dichloroethane	0.81	0.63	0.27	37 300	37 350	36 100	35 000	33 350
Dichloromethane	0.82	0.60	0.26	37 300	37 400	36 100	35 000	33 350
1,1,2,2-Tetrachloroethane	0.95	0.53	0.29	37 250	37 050	36 000	34 900	33 250

[a] Here and in Tables 3 and 4, π^* was taken from [2, 3], $f_1 = [(2n^2 + 1)/(n^2 + 2)]\{[(\varepsilon - 1)/(\varepsilon + 2)] - [(n^2 - 1)/(n^2 + 2)]\}$, $f_2 = (n^2 - 1)/(n^2 + 2)$, ε and n are taken from [10–13].

tion between the regression coefficient c in Eq. (1), the quantity μ_g, and the parameters c_1 and p. As nonspecifically solvating media we chose aprotic inert and aprotic protophilic solvents uncapable of hydrogen bonding with pyrazoles **I**–**V**.



$R^1 = NO_2$ (**I**), Me (**II**–**IV**), $CH{=}CH_2$ (**V**); R^2 = H (**I**, **III**), NO_2 (**II**), Me (**IV**, **V**); R^3 = H (**I**, **II**), NO_2 (**III**–**V**); R^4 = H (**I**–**III**), Me (**IV**, **V**).

Dipole moments of isomeric nitropyrazoles were measured in nonpolar and moderately polar solvents ($\pi^* < 0.6$) having no own dipole moment (Table 1). The results allow the solutes to be subdivided in two groups. In the first group (compounds **II**–**IV**, Table 1) μ_g somewhat increases in going from aprotic inert to aprotic protophilic solvents; this trend becomes more pronounced as the dipole moment of the solute increases. In the second group (pyrazoles **I** and **V**, Table 1) μ_g is independent of the solvent. Among the nitropyrazoles studied, 1-nitropyrazole has the lowest dipole moment, and 1-methyl-3-nitropyrazole, the highest dipole moment, which is consistent with the results of *ab initio* quantum-chemical calculations of μ_g of the free molecules.

Solvatochromism of the long-wave absorption band in the UV spectra of isomeric nitropyrazoles. The position of this band was determined in 35 sol-

Table 3. Solvatochromism of the long-wave absorption band in the UV spectra of isomeric nitropyrazoles **I**–**V** in aprotic protophilic solvents

Solvent	Solvent parameters			ν_{max}, cm^{-1}				
	π^*	f_1	f_2	**I**	**II**	**III**	**IV**	**V**
Triethylamine	0.14	0.07	0.24	37 900		37 100	36 100	34 300
Dibutyl ether	0.24	0.21	0.24	37 850	38 650	37 000	35 900	34 250
Diethyl ether	0.27	0.38	0.22	37 800	38 500	36 800	35 800	34 200
Dioxane	0.55			37 450	37 750	36 200	35 300	33 650
Ethyl acetate	0.55	0.49	0.23	37 500	37 800	36 300	35 400	
THF	0.58	0.55	0.25	37 400	37 700	36 200	35 300	33 500
Ethyl chloroacetate	0.70	0.65	0.25	37 350	37 150	36 000		
Acetonitrile	0.75	0.86	0.21		37 100	35 900	34 900	33 400
Propylene carbonate	0.83	0.88	0.25	37 300	36 850	35 700	34 650	33 250
Tetramethylurea	0.83	0.79	0.27	37 250	37 050	35 650	34 700	33 250
DMF	0.88	0.84	0.26	37 200	36 800	35 600	34 500	33 150
1-Methyl-2-pyrrolidone	0.92	0.81	0.28	37 150	36 700	35 500	34 500	33 100
DMSO	1.00	0.84	0.28	36 950	36 500	35 250	34 200	32 950

vents. Data on ν_{max} in aprotic inert and aprotic protophilic solvents are listed in Tables 2 and 3, respectively. Supplementary data are given in Table 4. Because N-substituted nitropyrazoles cannot form hydrogen-bonded complexes with aprotic protophilic solvents, we described the solvatochromism quantitatively in terms of the nonspecific solvation model [Eq. (1)]. The coefficients of Eq. (1) are listed in Table 5 which shows that only for 1-nitropyrazole and 1-vinyl-3,5-dimethyl-4-nitropyrazole the solvatochromism can be described by a single equation valid for all the aprotic media. For compounds **II**–**IV**, equations for the inert and protophilic solvents have different regression coefficients. To explain this fact, we considered three hypotheses.

The first hypothesis is based on considering the long-wave absorption band of isomeric nitropyrazoles, according to [14], as a sum of two (strong and weak) components. Both components are due to transitions with intramolecular charge transfer [14]. Therefore, the effect of the medium on the position of the overall maximum can be partially determined by the weak component. However, in this case extrapolation to the gas phase (ν_{gas}, Table 5) by Eq. (1) for all the five compounds should give different results depending on the solvent type (inert or protophilic). Actually, the difference in the estimated ν_{gas} values exceeds the experimental error (50 cm^{-1}) only for 1,3,5-trimethyl-4-nitropyrazole. That is, only in **IV** the complex nature of the long-wave absorption band plays a certain role.

The second hypothesis suggests a direct correlation, different for inert and protophilic solvents, between the dipole moment in the ground state and the regression coefficient *c*. This hypothesis is based on the fact that μ_g of pyrazoles **I** and **V** is independent of the solvent, whereas for **II**–**IV** such a dependence was observed experimentally (Table 1). As already noted, the solvatochromism of the first two compounds can be described by a common equation, whereas in the case of **II**–**IV** there are two specific cases (Table 5). Indeed, *c* does depend on μ_g, showing a nonlinear correlation (Fig. 1). However, the correlation includes both inert and protophilic solvents. Thus, the second hypothesis does not explain the specific

Table 4. Supplementary data on solvatochromism of nitropyrazoles **I**, **IV**, and **V**

Comp. no.	Solvent	Solvent parameters			ν_{max}, cm^{-1}
		π^*	f_1	f_2	
I	Dodecane	0.08	0.00	0.25	38 050
	Diisopropyl ether	0.27	0.33	0.23	37 900
	1-Bromoheptane	0.48	0.62	0.26	37 550
	Sulfolane	0.98	0.83	0.28	37 100
IV	Diisopropyl ether	0.27	0.33	0.23	35 750
	1,2-Dimethoxyethane	0.53	0.49	0.23	35 150
	Toluene	0.54	0.03	0.29	35 150
	Benzene	0.59	0.01	0.30	35 100
	Acetone	0.71	0.79	0.22	35 000
	Sulfolane	0.98	0.83	0.28	34 500
V	*p*-Xylene	0.43	0.01	0.29	33 700
	Benzene	0.59	0.01	0.30	33 550

Table 5. Quantitative characteristics of solvatochromism of nitropyrazoles **I**–**V**

Comp. no.	Solvent	μ^{25}, D	ν_0, cm^{-1}	c, cm^{-1}	r	s	n	ν_{gas},^{a} cm^{-1}	c_1, cm^{-1}	p, cm^{-1}	R	s	n^{b}
I	Inert	3.64^{c}	38 120 ± 20	–1000 ± 30	0.994	40	15	39 200	–1210 ± 50	–4 530 ± 170	0.994	40	$14^{d,e}$
	Protophilic	3.63^{f}	38 080 ± 40	–1040 ± 50	0.984	60	14	39 200	–830 ± 100	–5090 ± 460	0.977	90	14^{d}
	Inert and protophilic		38 110 ± 20	–1050 ± 30	0.990	60	29						
II	Inert	5.75^{c}	39 510 ± 30	–2640 ± 60	0.998	70	11	42 400	–3170 ± 220	–12 200 ± 700	0.988	170	11^{d}
	Protophilic	6.26^{f}	39 300 ± 70	–2850 ± 100	0.994	80	12	42 400	–2610 ± 280	–12 900 ± 107	0.987	180	12^{d}
III	Inert	4.35^{c}	37 660 ± 20	–1870 ± 40	0.997	60	13	39 700	–2290 ± 120	–8600 ± 400	0.991	100	13^{d}
	Protophilic	4.62^{f}	37 430 ± 40	–2100 ± 50	0.997	50	13	39 700	–1810 ± 120	–9 900 ± 500	0.992	100	13^{d}
IV	Inert	4.53^{c}	36 310 ± 30	–1640 ± 50	0.995	70	13^{g}	38 100	–1910 ± 100	–7 600 ± 300	0.992	80	$12^{d,e}$
	Protophilic	4.63^{f}	36 340 ± 50	–1970 ± 80	0.987	90	17	$38\,100^{h}$	–1320 ± 150	–9 400 ± 700	0.954	190	17^{d}
V	Inert	4.07^{c}	34 520 ± 30	–1490 ± 70	0.989	90	13	36 200	–1730 ± 140	–7 300 ± 500	0.979	120	13^{d}
	Protophilic	4.04^{f}	34 530 ± 50	–1580 ± 80	0.987	80	13	36 200	–1080 ± 120	–8 400 ± 600	0.972	150	13^{d}
	Inert and protophilic		34 520 ± 30	–1540 ± 50	0.990	80	26						

[a] (ν_{gas}) ν_{max} for the gas phase, estimated at $\pi^* = -1.1$ by Eq. (1). [b] Taking into account the estimated ν_{max} for the gas phase. [c] In tetrachloromethane. [d] Without 1-chlorobutane and dioxane. [e] Without 1-bromoheptane. [f] In benzene. [g] Without cyclohexane. [h] The value was obtained for aprotic inert media.

features of solvatochromism of **I**–**V** either. Furthermore, within a narrow group of protophilic solvents (xylene, benzene, dioxane) the measured dipole moment of **II**–**IV** is not always constant (Table 1). Hence follows that the regression coefficient *c* depends on a certain function which remains approximately constant within a set of related solvents. According to the theory, such a function can be the difference between the vectors of the dipole moments of the molecule in the ground and electronically excited states [Eq. (2)]—this is our third hypothesis.

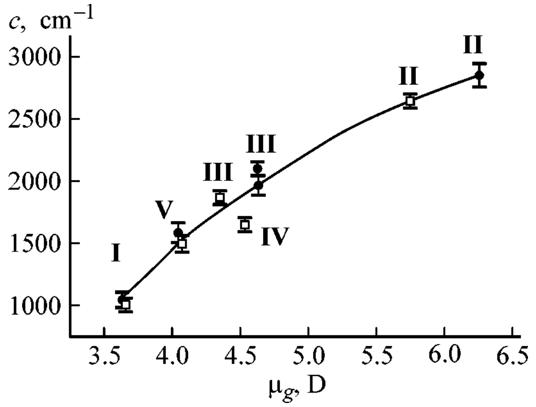


Fig. 1. Correlation between the regression coefficient *c* in Eq. (1) and the dipole moment of nitropyrazoles in the ground state μ_g: (*squares*) data for inert media (CCl_4 as example); (*circles*) data for protophilic media (benzene as example).

To describe the solvatochromism of isomeric nitropyrazoles with functions of universal interactions, we used the generalized solvent parameters ε and *n* from [10–13]. The values of f_1 and f_2 are given in Tables 2–4, and the parameters of Eq. (2), in Table 5. The data thus obtained allow us to examine the correlation between *c* and c_1 for each type of solvents separately. The correlation is shown in Fig. 2. In inert solvents *c* linearly correlates with c_1 (Fig. 2, curve *1*), with the straight line passing close to the origin. Hence, in inert media variation of *c* is governed by orientation interactions. In going from inert to protophilic solvents, the correlation between *c* and c_1 becomes nonlinear (Fig. 2, curve *2*), though *c* still tends to increase with increasing c_1. As seen from Table 5 (parameter *p*), in protophilic solvents a stronger effect on the spectrum is exerted by induction, polarization, and dispersion interactions, which is due to the presence in such solvents of readily polarizable nonbonding electron pairs and/or π-electron system. Thus, the third hypothesis also fails to describe the whole set of the experimental results.

It should be emphasized that in all cases, when using the functions of universal interactions, we had to exclude dioxane and 1-chlorobutane from the sample of solvents. The specific feature of dioxane may be due to the fact that the microscopic characteristics of the solvation shell formed by its molecules differ from the macroscopic characteristics of the solvent in the bulk. Within the framework of the Kamlet–Taft approach, these differences are taken into account automatically and are reflected in π^*. At the same time, the solvatochromism theory based on the Onsager–Batcher model fails to give the required specification. As for 1-chlorobutane, one of generalized parameters of this compound may have been determined insufficiently accurately. When describing the solvatochromism of 1,3,5-trimethyl-4-nitropyrazole, we excluded from consideration the values of ν_{max} corresponding to cyclohexane and 1-bromoheptane (Table 5), which is due to the fact that the long-wave band consists of components and the experimental error is therefore large. For example, instead of the ν_{max} value of 36 200 cm^{-1}, reported for the solution in cyclohexane in [6], after repeated measurements we obtained the average value of 36 300 cm^{-1}, which fits the suggested correlation much better. When using Eq. (2), the values of ν_{gas} (required to extend the variation range of ν_{max}) and f_2 were calculated by Eq. (1).

Thus, we failed to gain a deep insight into the effect of various types of solute–solvent interactions on the spectrum. This is due to the use of related objects and to large errors arising from the use of the functions of universal interactions. Nevertheless, we should ascertain that the regression coefficient at the solvatochromic parameter π^* is determined by the whole set of electrostatic and electrodynamic interactions between the solvent and solute (in the ground and electronically excited states) molecules. This evidently follows from the two-parameter equation

$$c = -(96 \pm 78) + (0.11 \pm 0.05)c_1 + (0.20 \pm 0.01)p;$$
$$R\ 0.987,\ s\ 70,\ n\ 10. \quad (3)$$

To the same c, there can correspond two pairs of c_1 and p (Table 5).

EXPERIMENTAL

The UV spectra of solutions of nitropyrazoles in various solvents were taken on a Specord UV-Vis spectrophotometer at 22–25°C. The dielectric permittivities of the solutions were measured with an Sh2-5 device (OKBA Joint-Stock Company, Angarsk) at 1 MHz. The dipole moments were calculated by the Higasi formula [15]. The substances were purified by recrystallization from organic solvents followed by double vacuum sublimation. *Ab initio* calculations of the dipole moments of nitropyrazoles in the 6–31G* basis were performed using the Gaussian 94 program package [16].

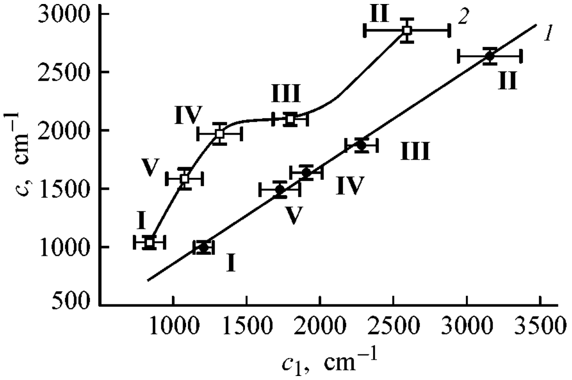


Fig. 2. Correlation between the regression coefficient c in Eq. (1) and parameter c_1 in Eq. (2): (*1*) inert and (*2*) protophilic solvents.

1-Nitropyrazole (**I**) was prepared by nitration of pyrazole by the procedure described in [17]. Pyrazoles **IV** and **V** were prepared according to [18].

1-Methyl-3-nitropyrazole (II). A 2-g portion of 3-nitropyrazole was dissolved in 30 ml of 1 N NaOH. The solution was heated to 30°C, and 1.64 ml of dimethyl sulfate was added dropwise. The mixture was kept for 1 h at 30°C. After cooling, the precipitate was filtered off, washed with cold water, and dried. Yield of **II** 0.83 g (39%), mp 86–87°C (cf. 85–86°C [19]).

1-Methyl-4-nitropyrazole (III). A 5.65-g portion of 4-nitropyrazole was dissolved in 60 ml of 1 N NaOH. The solution was heated to 30°C, and 4.8 ml of dimethyl sulfate was slowly added with stirring. After keeping for 1 h at 30°C, the mixture was cooled, and the precipitate was filtered off, washed with cold water, dried at 85°C, and recrystallized from ethanol. Yield of **III** 4.83 g (76%), mp 92–93% (cf. 91–92°C [20]).

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