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SOLUTE–SOLVENT INTERACTIONS IN ELECTRON DONOR–ACCEPTOR–SOLVENT SYSTEMS: PYRIDINE OR QUINOLINE DERIVATIVES, WITH MOLECULAR IODINE

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ABSTRACT

The thermodynamic characteristics (K, ΔH, ΔS) of the $D + I_2 = DI_2$ reaction, where D is an electron donor, have been obtained from the calorimetrically measured heats of solution of some quinolines and pyridines in pure solvents (*n*-hexane, cyclohexane, carbon tetrachloride, benzene, chlorobenzene), in iodine containing solutions, and from electronic spectra (340–600 nm) of amine iodine–solvent systems. The possibility of dissociation of the DI_2 complex into ionic species is discussed. The enthalpies of transfer of DI_2 complexes from cyclohexane to the other solvents have been calculated and interpreted in terms of solute–solvent interactions. For all the donors investigated the $\Delta H_{tr(DA)}$ values (A stands for acceptor) are comparable with the sum of the donor and acceptor ΔH_{tr} values and correlate fairly well with the polarity parameter, π^*, of the solvent.

INTRODUCTION

Solute–solvent interactions and their effect on equilibria in solutions containing non-ionic species have attracted much attention recently. Many attempts have been made to account in a quantitative way for the solvation of reactants. However, in most of the theoretical and almost all of the experimental studies only interactions of the solvent with the free donor (D) and acceptor (A) are taken into account. The interactions with the complex (DA) are, as a rule, neglected [1] or assumed to be a constant fraction of the sum of the solvation of the donor and acceptor, depending little on the choice of the solvent [2]. Such simplifications are a result of the lack of appropriate experimental data on the solvation of the DA complex itself. In fact, the determination of the thermodynamic characteristics of solvation would require the data on the complex formation in the vapour phase which, with present techniques, are rarely available for weak DA complexes. However, to discuss the relative solvent effect on complex solvation one can

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use the thermodynamic functions of transfer of the complex, $\Delta H_{\mathrm{tr(DA)}}$ from an arbitrarily chosen reference (r) solvent to a given solvent (s)

$$\begin{tikzcd}
\mathrm{D(r)} \arrow[d, "\Delta H_{\mathrm{tr(D)}}"'] & + & \mathrm{A(r)} \arrow[d, "\Delta H_{\mathrm{tr(A)}}"'] \arrow[r, rightleftharpoons, "\Delta H_{\mathrm{DA(r)}}"] & \mathrm{DA(r)} \arrow[d, "\Delta H_{\mathrm{tr(DA)}}"] \\
\mathrm{D(s)} & + & \mathrm{A(s)} \arrow[r, rightleftharpoons, "\Delta H_{\mathrm{DA(s)}}"] & \mathrm{DA(s)}
\end{tikzcd} \qquad (1)$$

However, such data for DA complexes are also extremely scarce, because most thermodynamic results on such complexes have been obtained by spectroscopic techniques. These techniques give valuable results in determining the equilibrium constants, but much less reliable enthalpic data which makes them less suitable for calculating thermodynamic cycles [3].

In this paper we present the results of calorimetric and spectrophotometric investigations on the complex formation and solute–solvent interactions in solutions containing quinoline/pyridine derivatives and molecular iodine in *n*-hexane, cyclohexane, carbon tetrachloride, benzene and chlorobenzene. These systems represent different groups of non-hydrogen bonding media (aliphatic, alicyclic, and aromatic non-chlorinated and chlorinated hydrocarbons).

EXPERIMENTAL

The solvents were purified by standard methods and fractionally distilled. The liquid amines (Fluka AG Bush G, Switzerland, pure) were dried by prolonged standing over KOH pellets and distilled from molecular sieves (4 Å) in vacuo. Solid 2-aminopyridine (pure, Roanal Budapest) was recrystallized from benzene–hexane [4]. Iodine (pure grade) was resublimed twice with KI.

Calorimetric measurements

The enthalpies of solution or dilution of the amines and the enthalpies of complex formation were determined under moisture-free conditions using a "constant-temperature environment" calorimeter [5] (100-cm^3 glass vessel; 1-cm^3 teflon ampoule with a thin glass bottom crushed to start the reaction). The heats of complex formation, ΔH_{DA}, were calculated as $\Delta H_{\mathrm{DA}} = (q_1 - q_2)/n$, where q_1 is the measured enthalpy of solution of ca. 1 cm^3 of the pure amine in iodine solution (10^{-4}–10^{-3} mol dm^{-3}) in the given solvent (ca. 90 cm^3), q_2 is the heat of solution of the pure amine in the same solvent determined separately for the same conditions and with the same final amine concentrations (10^{-3}–10^{-1} mol dm^{-3}), and n is the number of moles of the complex in the solution calculated from spectrophotometric measurements. The measurements in hexane and cyclohexane were made with concentrated amine solutions in the given solvent (1–3 mol dm^{-3}) instead of pure amine because of the high endothermic heats of solution of pure amines in aliphatic solvents [5]. The heat of dilution of the iodine solution to the final concentration was found to be negligible. The heat capacity of the calorimetric system was determined electrically with an accuracy of 0.2%.

Spectrophotometric measurements

The electronic spectra (340–600 nm) were measured on a Beckmann 5270 UV spectrophotometer in thermostatted (±0.1 K) stoppered cells. The modified Ketelaar equation, which accounts for iodine absorption, was applied to absorbancies in the region of the perturbed iodine band. The equilibrium-constant values were calculated for five analytical wavelengths near the band maximum. The calculations were performed for at least three sets of solutions (not less than six solutions in the set). In every set the acceptor concentration (ca. 10^{-4} mol dm^{-3}) was kept constant, while the donor concentration was varied from 10^{-2} to 10^{-3} mol dm^{-3}.

The K_{DA} and ΔH_{DA} values listed in Table 1 are averages of two to five independent sets of measurements. The uncertainty limits were calculated as standard deviations of the mean values.

Conductometric measurements

Conductometric measurements were made using a standard conductivity cell with platinum electrodes and a Beckman RC-18A conductometer at 1 kHz and 3 kHz frequencies at a constant temperature (298.2 ± 0.01 K). The cell constant was 0.0251 cm^{-1}. All solutions were freshly prepared just before the first measurement of the given property.

RESULTS AND DISCUSSION

All the data listed in Table 1 refer to the reaction of complex formation involving electrically neutral species

$$D + I_2 = DI_2 \qquad (2)$$

In solvents of low polarity, equilibrium (2) dominates over other possible equilibria which are solute–solvent interactions. In polar media, however, reaction (2) is followed by the processes of ionic dissociation of the DI_2 complex [6,7]:

$$DI_2 = DI^+I^- \qquad (3)$$

$$DI^+I^- = DI^+ + I^- \text{ or } 2DI^+I^- = D_2I^+ + I_3^- \qquad (4)$$

TABLE 1

Thermodynamic characteristics of the reaction $D+I_2=DI_2$ (where D is a quinoline or pyridine derivative) and the heats of transfer of the electron donor (D) and EDA complex (DA) from cyclohexane to weakly and moderately polar solvents at 298.2 K (in kJ mol^{-1}) [a]

Solvent	$K_{(DA)}$	$-\Delta G_{(DA)}$	$-\Delta H_{(DA)}$	$-\Delta S_{(DA)}$ $\times 10^3$	$-\Delta H_{tr(D)}$	$-\Delta H_{tr(DA)}$
Quinoline, pK_a 4.87						
n-Hexane	122±6	11.90±0.03	31.8±1.2 [e]	67±5	0.6 ±0.3	1.1
Cyclohexane	114±5	11.73±0.10	31.3±1.4 [e]	66±5	0	0
CCl_4	86±2	11.03±0.06	30.1±1.0 [e]	64±6	7.3 ±0.1	6.1
Benzene	60±2	10.14±0.08	25.5±1.0 [e]	52±4	7.9 ±0.3	8.6
Chlorobenzene	143±3	12.30±0.05	31.3±1.2 [e]	64±3	8.3 ±0.3	12.7
6-Methylquinoline, pK_a 4.92						
n-Hexane	152±3	12.45±0.04	40.4±1.2	94±4	1.8 ±0.3	3.2
Cyclohexane	166±6	12.66±0.09	39.0±1.7 [e]	88±6	0	0
CCl_4	131±6	12.08±0.09	32.7±1.0	69±4	8.1 ±0.2	1.8
Benzene	75±0	10.70±0	36.0±1.0	85±3	8.3 ±0.2	11.8
Chlorobenzene	205±3	13.19±0.03	41.5±0.5 [e]	95±2	9.6 ±0.3	16.5
Isoquinoline, pK_a 5.47 [b]						
n-Hexane	198±3	13.10±0.04	37.0±0.3	80±2	1.8 ±0.2	2.2
Cyclohexane	187±3	12.96±0.04	36.6±0.2	79±1	0	0
CCl_4	149±5	12.40±0.08	32.9±0.5	69±3	9.6 ±0.1	5.9
Benzene	109±1	11.62±0.02	31.9±0.1	68±1	10.2 ±0.1	12.0
Chlorobenzene	260±5	13.78±0.04	37.7±0.3	80±2	10.2 ±0.1	15.7
Pyridine, pK_a 5.20 [c]						
n-Hexane	133±4	12.12±0.07	35.7±0.9	79±3	0.19	1.3
Cyclohexane	129±4	12.04±0.08	34.6±0.1	76±1	0	0
CCl_4	105±3	11.53±0.07	30.4±0.9	63±3	7.51	3.3
Benzene	82±3	10.92±0.09	29.9±0.1	64±1	8.24	10.0
Chlorobenzene	182±6	12.89±0.08	33.5±0.6	69±3	8.29	11.6
2-Methylpyridine, pK_a 5.96 [c]						
n-Hexane	181±9	12.88±0.12	37.6±0.1	83±1	0.81	1.4
Cyclohexane	186±8	12.95±0.1	37.0±0.9	81±3	0	0
CCl_4	143±2	12.30±0.03	36.1±0.6	80±2	7.08	6.2
Benzene	100±4	11.41±0.09	34.1±0.5	76±2	6.51	10.1
Chlorobenzene	249±4	13.67±0.04	36.1±0.6	75±2	7.55	11.1
2,4-Dimethylpyridine, pK_a 7.25 [b]						
n-Hexane	390±5	14.78±0.03	37.7±0.4	77±2	1.6 ±0.2	1.0
Cyclohexane	364±1	14.61±0.01	38.3±0.1	80±1	0	0
CCl_4	284±3	14.00±0.03	36.6±0.5	76±2	9.0 ±0.2	7.3
Benzene	228±3	13.45±0.03	36.0±0.3	76±2	7.2 ±0.1	11.4
Chlorobenzene	575±9	15.74±0.04	41.0±0.1	85±1	8.8 ±0.1	15.9
2,6-Dimethylpyridine, pK_a 6.64 [b]						
n-Hexane	72±1	10.60±0.04	34.8±0.1	81±1	0.2 ±0.1	0.6
Cyclohexane	75±2	10.70±0.06	34.5±0.2	80±1	0	0
CCl_4	58±2	10.06±0.07	32.5±0.1	75±1	6.8 ±0.1	4.8
Benzene	42±1	9.26±0.06	28.6±0.3	65±2	5.1 ±0.2	5.7
Chlorobenzene	85±3	11.01±0.08	35.6±0.5	82±2	6.8 ±0.2	12.3

TABLE 1 (continued)

Solvent	$K_{(DA)}$	$-\Delta G_{(DA)}$	$-\Delta H_{(DA)}$	$-\Delta S_{(DA)}$ $\times 10^3$	$-\Delta H_{tr(D)}$	$-\Delta H_{tr(DA)}$
2-Aminopyridine, pK_a 6.86						
n-Hexane	276 ± 6	13.92 ± 0.06	41.2 ± 2 [d]	91 ± 1	–	–
Cyclohexane	273 ± 4	13.90 ± 0.03	$38.0 + 2$ [d]	81 ± 1	0	0
CCl_4	195 ± 3	13.06 ± 0.04	33.7 ± 3 [d]	69 ± 2	8.3 ± 3	4
Benzene	148 ± 3	12.38 ± 0.05	32.3 ± 0.5	66 ± 2	12.7 ± 2	13.4
Chlorobenzene	302 ± 4	14.15 ± 0.03	37.6 ± 0.5	79 ± 2	12.2 ± 2	16.2

[a] Heats of transfer of the acceptor $\Delta H_{tr(I_2)}$, are from ref. 16.
[b] Ref. 5
[c] Ref. 19.
[d] Ref. 20.
[e] Spectrophotometric methods.

The above reactions have been extensively studied for pyridine as a donor in pure pyridine ($\epsilon = 12.3$) [6,7] and in polar solvents ($\epsilon > 10$) [7]. Such ionization processes must also be taken into account when solvents of medium

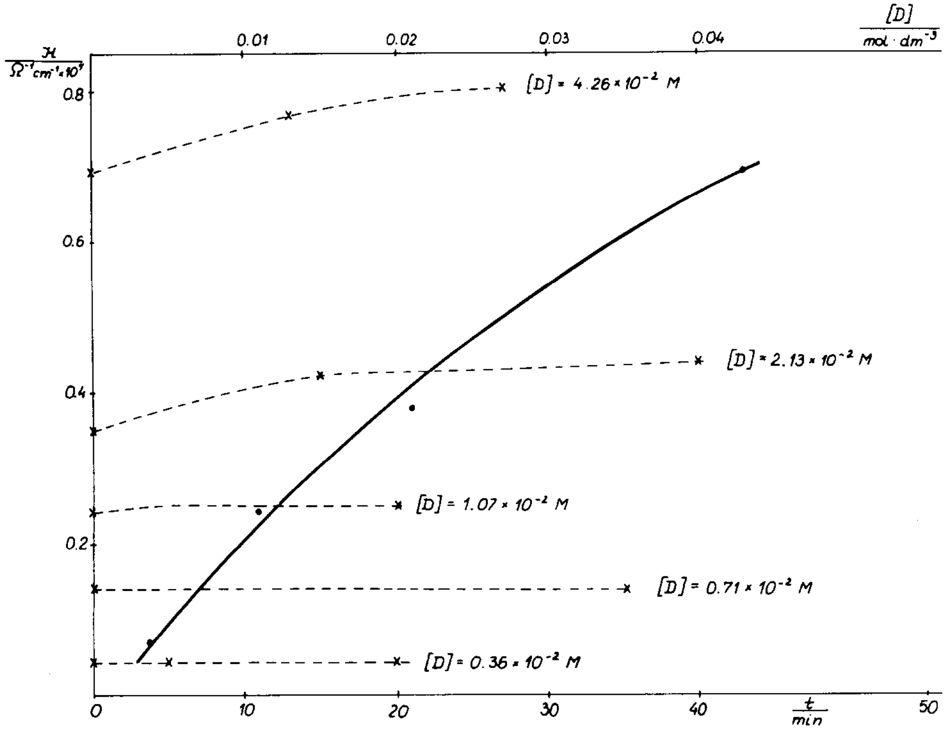

Fig. 1. The electrical conductivity of the 6-methylquinoline–iodine–chlorobenzene solutions at 298.2 K; $[I_2] = \text{constant} = 9.05 \times 10^{-4}$ mol dm^{-3}. ×, Dependence on the time; ●, dependence on the donor concentration.

polarity are to be used. Fortunately, reaction (3) is slow, particularly at low concentrations of the reactant, so that at favourable conditions the presence of ionic species may be neglected. The validity of such an assumption must, however, be tested by spectrophotometric (fine isosbestic points, time-independent absorptivity in the UV–visible region) and conductometric measurements. This has been done for the systems reported in this work. As expected, solutions in *n*-hexane, cyclohexane, benzene and carbon tetrachloride ($\epsilon < 2.3$) showed no evidence of ionic processes, even for the strongest donors in the series ($pK_a > 7$) where the percentage of dative structure, and thus the tendency to ionization, should be comparatively high. The electrical conductivity was constant within the experimental error and its value was below 10^{-7} Ω^{-1} cm^{-1}; the absorptivities of the solutions kept in the dark did not change by more than 1.5% after 24 h.

The ionization processes can be observed in chlorobenzene ($\epsilon = 5.62$). The measured conductivity showed an appreciable dependence on time, and on the concentrations of the reactants (typical plots are shown in Fig. 1). However, at the donor and acceptor concentrations used in this study, the conductivities measured after 1 or 2 h were low enough (of the order 10^{-7} Ω^{-1} cm^{-1}) to neglect ionic species in calculating the thermodynamic characteristics of reaction (2). This conclusion can be supported by the presence

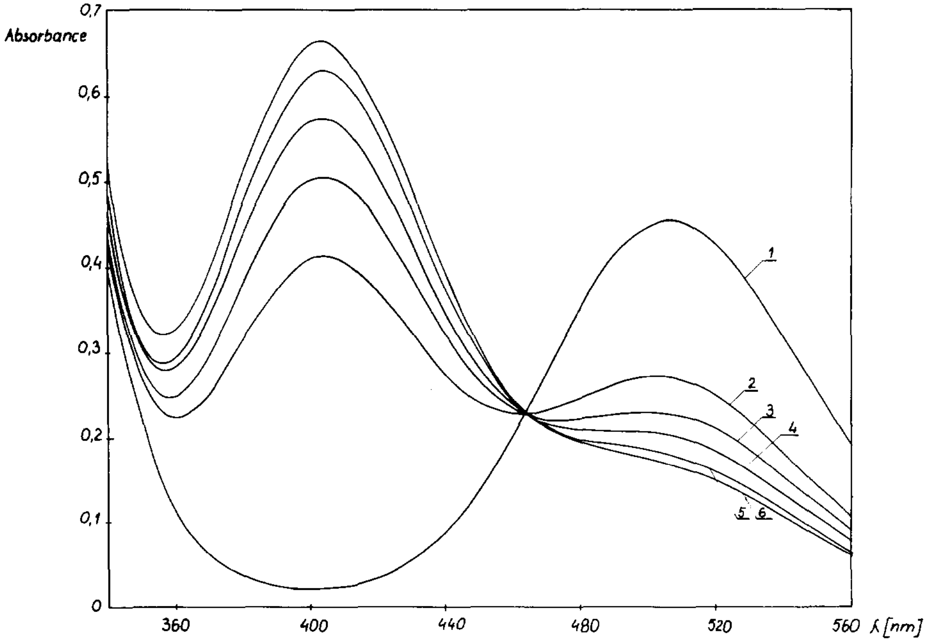

Fig. 2. The electronic spectra of 6-methylquinoline–molecular iodine–chlorobenzene solutions at 298.2 K; $l = 2$ cm; $[I_2] = 2.385 \times 10^{-4}$ mol dm^{-3}. $[6\text{-MeQ}] \times 10^{-3} = 0$ (1); 3.702 (2); 5.553 (3); 7.404 (4); 9.255 (5); 11.106 (6).

of clear isosbestic points in the electronic spectra [Fig. 2] and negligible changes in absorptivity; in particular, no increase in the intensity in the region of the absorption characteristic of the ionic species (below 395 nm [6]) was observed.

The values of K, ΔH and ΔS for reaction (2) are listed in Table 1. For all the donors investigated the K and ΔH_{DA} values varied with the reaction medium in the same order: *n*-hexane ≃ chlorobenzene > cyclohexane > carbon tetrachloride > benzene. Within the series of low-polarity solvents ($\epsilon < 2.3$), the ln K and ΔH_{DA} values decreased with increasing polarity of

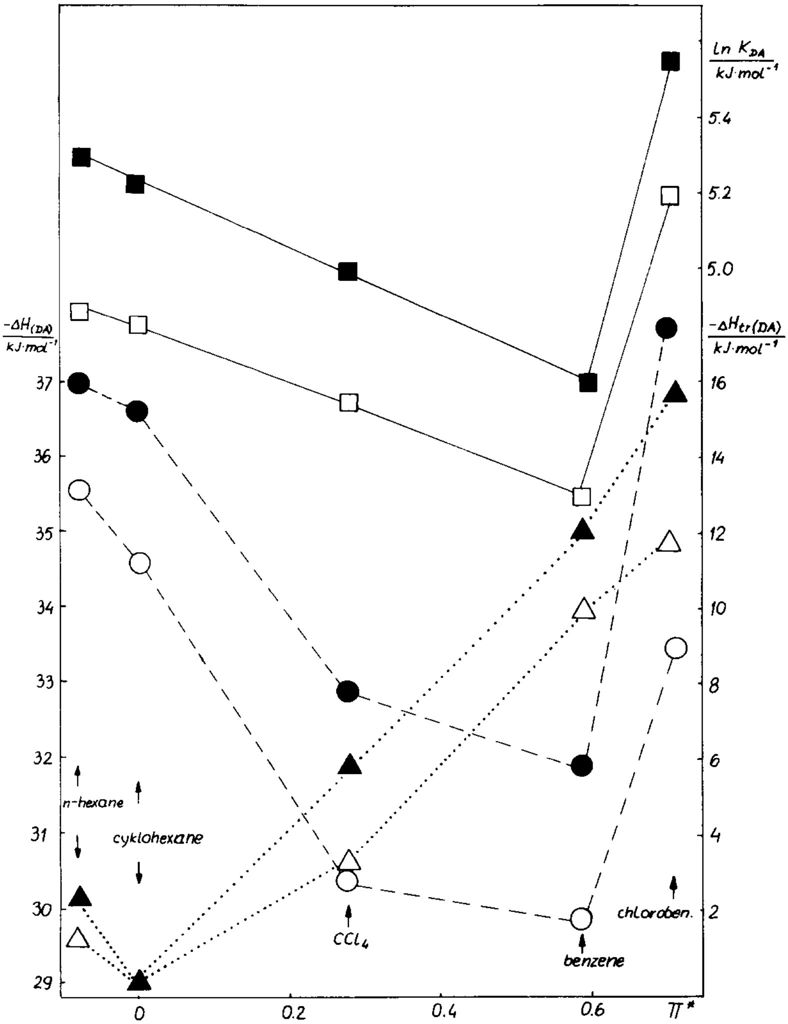

Fig. 3. Plots of ln K_{DA} (■, □), enthalpy of complex formation $\Delta H_{(DA)}$ (●, ○) and enthalpy of transfer of the DI_2 complex from cyclohexane $\Delta H_{tr(DA)}$ (▲, △) versus the polarity parameter of the solvent π^*. ■●▲, Isoquinoline; □○△, pyridine.

the solvent, whatever the polarity scale [8] used. The best, nearly linear, correlation was obtained by plotting ln K versus the polarity parameter, π^* [9] (Fig. 3). The correlation of the ΔH_{DA} values with π^* is less satisfactory because of the greater scatter of the experimental points (the lines connecting the experimental points in Fig. 3 have no physical meaning, they only make the plot more readable). For the sake of clarity, Fig. 3 shows only the results for isoquinoline and pyridine as donors, the plots for other systems are similar.

The whole sequence of the K and ΔH_{DA} values cannot be correlated with any physical property of the solvent or any arbitrary commonly accepted characteristics of the medium, because no scale of solvent polarity or reactivity allows for the dramatic increase in the K and ΔH_{DA} values on passing from weakly polar media to chlorobenzene and other polar chlorinated aromatics investigated in previous work (bromobenzene, *ortho*-dichlorobenzene [10]). As discussed above the peculiar behaviour of the complexes in chlorobenzene cannot be explained by ionic processes provoked by the polar medium. Nor should it be ascribed to specific solute–solvent interactions which are the most frequently encountered cause of the deviations from the linear relationships between the stability of electron donor–acceptor complexes and the solvent polarity [11]. To account for such significant deviations, the specific interactions need to be comparatively strong and they should stabilize the complex or destabilize its constituents with respect to other solvents. However, no strong specific interactions of the complex with chlorobenzene could be anticipated: the π–π interactions of the aromatic rings should be rather weak and they are not likely to be much different from those exhibited by the donor itself. Moreover, such π–π interactions should be comparable for benzene and chlorobenzene as solvents, which is also the case for the very weak acceptor–solvent interactions (K_{DA} values measured in this work for the iodine–solvent complex in *n*-hexane are 1.2 and 0.7 dm^3 mol^{-1} for benzene and chlorobenzene, respectively). Thus the main cause of the dramatic change in the K_{DA} and $-\Delta H_{DA}$ values on passing from benzene to chlorobenzene should be the non-specific solvation. Increasing polarity of the medium enhances this type of solvation of the polar complexes more rapidly than the solvation of a less polar donor and acceptor (dipole moments of the pyridine and quinoline derivative–iodine complexes exceed the sum of the dipole moments of the donor and acceptor by more than 100% [12–15]). However, the donor and acceptor must be partly desolvated as a result of complex formation. The last process most probably dominates the first one in weakly polar solvents, while the reverse is true in the moderately polar chlorobenzene.

In an attempt to obtain some insight into solvation relationships in the investigated donor–acceptor solvent systems, we calculated the enthalpies of transfer of the constituents and that of the complex itself from the weakly solvating medium of cyclohexane to different solvents used in this work. The $\Delta H_{tr(D)}$ values were derived from the heats of solution of the amines determined calorimetrically in an experiment parallel to the measurements of the enthalpies of complex formation; the $\Delta H_{tr(I_2)}$ values were taken from ref. 16 (it is essential for such studies that the measurements should be performed under the same conditions, but the error in $\Delta H_{tr(I_2)}$ is cancelled out by comparisons between different donor systems); the $\Delta H_{tr(DA)}$ values were calculated from scheme (1).

The results show that the enthalpies of transfer of the donor (Table 1) do not follow any polarity scale of the solvent, the values of $\Delta H_{tr(D)}$ being nearly equal for CCl_4, benzene and chlorobenzene and much more exothermic than for *n*-hexane. This behaviour has been explained by weak specific solute–solvent interactions involving the π-electron system of the aromatic ring and the frontier orbitals of benzene, chlorobenzene and carbon tetrachloride [17]. It is clear that the $\Delta H_{tr(I_2)}$ values reflect the specific charge-transfer interactions of iodine with benzene and chlorobenzene (for *n*-hexane, cyclohexane, carbon tetrachloride, benzene and chlorobenzene they are 0, 0, 0, −6.5 and −4.4 kJ mol^{-1}, respectively) and do not follow the polarity of the medium. However, for the complex itself, a fairly good correlation of $\Delta H_{tr(DA)}$ (Table 1) with the polarity parameter, can be found (Fig. 3, triangles) for most of the systems investigated. The scatter of the points in the plot may be due to the experimental error which is greater for $\Delta H_{tr(DA)}$ (a quantity calculated from a thermodynamic cycle) than for the other quantities discussed above. There is, however, one deviation systematically observed in the $\Delta H_{tr(AD)}$ versus π^* plots: the experimental points for *n*-hexane lie higher than those for cyclohexane. For each particu-

TABLE 2

The ratio of the enthalpy of transfer of the DA complex to the sum of the enthalpies of transfer of the donor (D) and acceptor (iodine) from cyclohexane to the solvent: $\alpha \equiv \Delta H_{tr(DA)}/(\Delta H_{tr(D)} + \Delta H_{tr(I_2)})$

Donor	α			
	n-Hexane	CCl_4	Benzene	Chlorobenzene
Quinoline	~ 2	0.8	0.6	1.0
6-Methylquinoline	~ 2	0.2	0.8	1.2
Isoquinoline	~ 1	0.6	0.7	1.1
Pyridine	~ 6	0.4	0.7	0.9
2-Methylpyridine	~ 2	0.9	0.8	0.9
2,4-Dimethylpyridine	~ 1	0.8	0.8	1.2
2,6-Dimethylpyridine	~ 3	0.7	0.5	0.9
2-Aminopyridine	–	0.5	0.7	1.0
Average value	> 1	0.6	0.7	1.0

lar donor the difference is within the experimental error; however, it must reflect real differences in solvation between the two solvents because positive $\Delta H_{\text{tr(DA)}}$ values occur for each donor system. It is interesting that the same behaviour can be observed for the enthalpies of transfer of the donors, while ΔH and ΔG values for reaction (2) behave "regularly".

In an attempt to estimate the relationship between the solvation of the complex and its constituents we calculated the α factor defined by Christian [18]

$$\alpha \equiv \frac{\Delta H_{\text{solv(DA)}}}{\Delta H_{\text{solv(D)}} + \Delta H_{\text{solv(A)}}} \simeq \frac{\Delta H_{\text{tr(DA)}}}{\Delta H_{\text{tr(D)}} + \Delta H_{\text{tr(A)}}} \tag{5}$$

(Table 2). In spite of the error involved in such calculations, the results seem to be reasonable because they are based on measurements for eight independent systems. The results show that formation of the DI_2 complex in weakly polar solvents results in a decrease in the overall solvation of the solutes with respect to the situation in cyclohexane ($\alpha < 1$), while in chlorobenzene both processes (an increase in non-specific solvation and "squeezing" of the solvent molecules from the solvation spheres of the donor and acceptor) are nearly balanced ($\alpha \simeq 1$).

The striking feature of the results presented in Tables 1 and 2 is that all the eight donor systems show the same dependence on the solvent. They also show comparable enthalpies of transfer of the donors and the complexes to the same solvent, irrespective of the steric and electronic properties of the donors used. These facts indicate that the differences in the solvation of various donor–acceptor systems of the series investigated are negligible. This, in turn, suggests that solvation of the substituents in the donor ring has a local character. Such a conclusion explains why the pyridine derivative–I_2 solvent systems very well obey the linear free energy relationships found for donors with different substituents and various basicities ($pK_a = 0.72$–7.3) [10]. No particular behaviour has been observed for the increased π-electron system of the donor (quinolines). In all the systems investigated the solute–solvent interactions involving π electrons seem to be rather weak in comparison with non-specific solvation. This type of interaction seems to be responsible not only for the solvation of the complexes but also for the solvation of the pyridines themselves [17].

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