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STUDY OF THE SPECTRAL BEHAVIOUR OF MOLECULAR COMPLEXES OF PICRIC ACID AND HEXANITROBIPHENYLAMINE WITH SCHIFF BASES FORMED FROM SALICYLALDEHYDE AND ALKYL-AMINES, POLYMETHYLENEDIAMINES, SULPHONAMIDES AND AMINOPYRIDINES

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Molecular complexes of picric acid and hexanitrobiphenylamine with Schiff bases (SB) derived from salicylaldehyde and alkylamines, polymethylenediamines, sulphonamides and aminopyridines, with formulae SB.A or $SB.A_2$ (A = acceptor molecule), were prepared and characterized through UV, visible, IR and ^1H NMR spectroscopy. Picric acid and hexanitrobiphenylamine act as acceptor molecules and bind to the aromatic rings of the donors via intermolecular $\pi - \pi$ charge-transfer interactions.

Pfeiffer [1] suggested that the interactions between the components in a molecular complex could be regarded as interactions between particular force fields to be assigned to certain regions of the molecules. For example, when an aromatic amine is complexed with a polynitrophenol, for instance picric acid, PA, two types of force field must be considered: one type of force field produces an acid-base

interaction (proton transfer, PT), and the other type an electron donor-acceptor interaction (charge transfer, CT).

The preparation of PA complexes through the use of different solvents, and the isomerism of CT-PT complexes, were extensively studied by Matsunaga et al. [2-4]. Issa et al. [5-6] pointed out that, in the molecular complexes of benzylidene derivatives and PA, the occurrence of CT, PT and n-π interactions is possible.

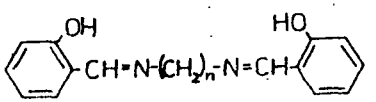
As part of a continuing survey of the spectral characteristics of Schiff bases (SB) [7-10] and of their molecular complexes [11, 12], we have examined the ability of the SB derived from salicylaldehyde with alkylamines, [13], polymethylenediamines [9], sulphonamides [14] and aminopyridines to form molecular complexes with different acceptor molecules. We now report the preparation and the UV, visible, IR and ^{1}H NMR spectra of molecular complexes of SB of types I, II, III and IV as donors with PA or hexanitrobiphenyl-amine (dipicrylamine, DPA) as acceptor molecule (see Table I).

Experimental

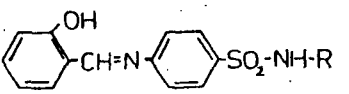
The SB were synthetized as described previously [13-15]. The complexes were prepared by mixing hot methanolic solutions of donor and acceptor in 1:1 and 1:2 mole ratios. On cooling, crystalline products separated out, which were filtered off and washed with cold ethanol and ether. The m.p.s. and the analytical data on the complexes prepared are listed in Table II. The SB with even numbers of methylene groups form crystalline products with PA more easily and faster, but in the case of longer chains (n > 8) no crystal-

Table I

The Schiff bases studied and the numbering of their molecular complexes in the text



I (PA, DPA)

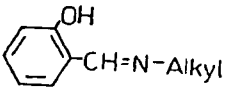


II (PA)

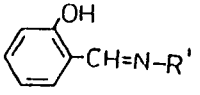
No.	n	Comp.	No.	n	Comp.	No.	R*
1	0	1:1	5	5	1:1	8	$-C_6H_7N_2$
1a		1:2	5a		1:2	9	$-C(O)NH_2$
2	2	1:1	6	6	1:1	10	$-C_5H_5N_2O$
2a		1:2	6a		1:2	11	$-C_6H_7N_2$
3	3	1:1	7	8	1:1	12	$-C_5H_5N_2O$
3a		1:2	7a		1:2	13	$-C_6H_7N_2O_2$
4	4	1:1				14	$-C_5H_6NO$
4a		1:2				15	$-C_4H_4NS$

* The parent sulphonamides: 8: 2-(4'-aminophenylsulphonyl)-amino-4,6-dimethylpyrimidine; 9: (4'-aminophenylsulphonyl)-carbamide; 10: 6-(4'-aminophenylsulphonyl)-amino-3-methoxy-pyridiazine; 11: 6-(4'-aminophenylsulphonyl)-amino-2,4-dimethylpyrimidine; 12: 2-(4'-aminophenylsulphonyl)-amino-5-methoxypyrimidine; 13: 6-(4'-aminophenylsulphonyl)-amino-2,4-dimethoxypyrimidine; 14: 5-(4'-aminophenylsulphonyl)-amino-3,4-dimethyl-1,2-oxazole; 15: 5-(4'-aminophenylsulphonyl)-amino-3-methyl-1,4-thiazole.

Table I (Continued)



III (DPA)



IV (DPA)

No.	Alkyl	No.	Alkyl	No.	R'**
16	CH_3	22	$n\text{-}C_5H_{11}$	28	$-C_5H_4N$
17	C_2H_5	23	$i\text{-}C_5H_{11}$	29	$-C_5H_4N$
18	$n\text{-}C_3H_7$	24	C_6H_{13}	30	$-C_5H_4N$
19	$i\text{-}C_3H_7$	25	C_8H_{17}	31	$-C_5H_3N(CH_3)$
20	$n\text{-}C_4H_9$	26	$C_{10}H_{21}$	32	$-C_5H_3N(CH_3)$
21	$i\text{-}C_4H_9$	27	$C_{12}H_{25}$	33	$-C_5H_3N(CH_3)$

** Amines used: 28: 2-aminopyridine; 29: 3-aminopyridine; 30: 4-aminopyridine; 31: 2-amino-3-methylpyridine; 32: 2-amino-4-methylpyridine; 33: 2-amino-5-methylpyridine; 34: 2-amino-6-methylpyridine.

line product can be isolated. The DPA complexes of salicylidene-alkylamines (16-27) were investigated only in solutions prepared with different mole ratios.

The UV and visible spectra were recorded on a SPECORD UV-VIS spectrophotometer in spectroscopically pure solvents at room temperature. The IR measurements were carried out on a ZEISS SPECORD M80 instrument in KBr discs. The ^1H NMR spectra were obtained on a BRUKER FT80 instrument in DMSO-d_6, using TMS as internal standard***.

*** The author is grateful to Dr. G. Horváth (CHINOIN) for the ^1H NMR and IR measurements.

Table II

Analytical data on the molecular complexes prepared

No.	Col*	M.P.**	C %		H %	
			Calcd.	Found	Calcd.	Found
1	LY	170 - 173	51.18	51.07	3.12	3.20
1a	LY	193.5 - 194.5	44.71	44.06	2.60	2.33
2	LY	113.5 - 114.0	53.12	52.44	3.85	4.00
2a	LY	170 - 172	46.29	47.82	3.06	2.84
3	O	147 - 149	54.01	53.87	4.14	4.05
3a	LY	177 - 179	47.03	46.77	3.27	3.09
4	O	175 - 176	54.86	54.66	4.41	4.31
4a	LY	195 - 196	47.75	47.51	3.47	3.33
5	O	123.5 - 124.5	55.66	55.33	4.67	4.56
5a	LY	164.5 - 165.5	48.44	48.37	3.67	3.51
6	LY	155 - 156	56.42	56.40	4.92	4.77
6a	LY	175 - 176	49.11	49.01	3.86	3.88
7	LY	153.5 - 154.5	57.83	57.77	5.37	5.31
7a	LY	161 - 162	50.37	49.98	4.23	4.21
8	LY	113 - 114	44.29	44.08	2.88	2.72
9	BY	139 - 140	40.16	39.88	2.46	2.33
10	LY	160 - 161	42.76	41.64	2.63	2.53
11	O	106 - 107	44.29	44.08	2.88	2.58
12	LY	180 - 181	42.76	42.59	2.63	2.55
13	O	74 - 75	42.67	42.55	2.77	2.59
14	O	77 - 78	43.43	43.09	2.79	2.68
15	O	119 - 120	41.88	41.66	2.55	2.52
28	R	177.0 - 177.5	40.16	39.86	1.87	2.01
29	SC	186 - 187	45.32	45.85	2.37	2.50
30	R	199.5 - 200.0	40.16	39.54	1.87	1.94
31	SC	197 - 198	40.75	40.28	2.03	1.96
32	BR	196 - 197		40.33		1.88
33	SC	175 - 176		40.25		1.92
34	SC	186 - 187		39.98		2.00

* Y: yellow; R: red; SC: scarlet; BR: brownish-red; O: orange; LY: lemon-yellow; BY: brownish-yellow

** uncorrected values.

Results and Discussion

A) *PA complexes 1-7 (1:1) and 1a-7a (1:2).* Upon complex formation, the spectra of the SB [15] change considerably (Table III); the 240-320 nm bands become indistinct, and only inflections appear (Fig. 1/2). In the range 350-420 nm,

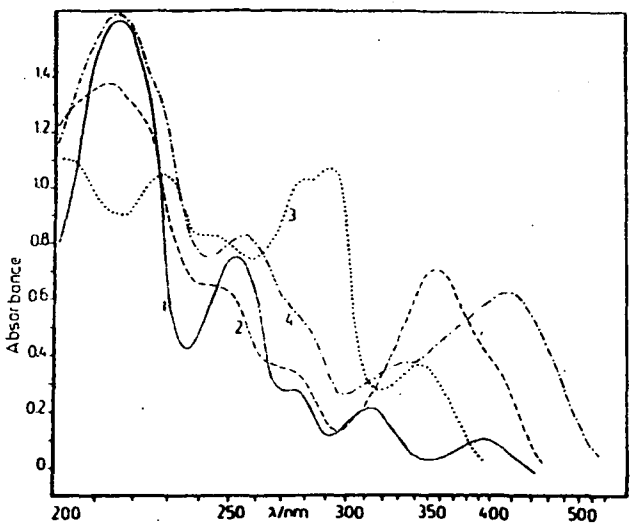


Figure 1.: UV and visible spectra of the SB(n=5) (1), $c=2.09.10^{-4}$; 5a (2), $c=3.90.10^{-4}$; 5a in cc sulphuric acid (3), $c=3.85.10^{-4}$; the 1:1 DPA complex (4), $c=2.12.10^{-6}$ mol/dm^3. Solvent (1, 2, 4): methanol; d=0.1 cm.

a medium-intensity, broad band appears with an inflection on the long-wavelength side. In chloroform (and in other solvents not forming hydrogen-bonds), the spectral struc-

Table III

Spectral data on the PA complexes

No.	λ/nm and log ε measured in CH_3OH					
1	220(4.63)	~240	-	295(4.57)	356(4.61)	~420
1a	220(4.86)	~240	-	294(4.73)	354(4.81)	~420
2	415(4.69)	~230	257(4.39)	~280	354(4.39)	~400
2a	214(4.75)	~230	252(4.50)	~280	353(4.48)	~400
3	216(4.60)	~230	~245	~280	351(4.39)	~410
3a	214(4.72)	~230	~240	~280	352(4.58)	~410
4	214(4.82)	~225	253(4.51)	~278	351(4.44)	~400
4a	213(4.79)	~225	~255	274(4.49)	353(4.53)	~400
5	214(4.75)	~225	~250	~280	355(4.51)	~400
5a	211(4.73)	~225	-	275(4.48)	355(4.63)	~400
6	214(4.77)	~225	257(4.46)	274(4.45)	352(4.41)	~400
6a	211(4.83)	~225	-	274(4.63)	352(4.64)	~400
7	212(4.59)	~225	-	274(4.33)	354(4.44)	~400
7a	213(4.79)	~225	-	275(4.59)	354(4.66)	~400
8	204(4.79)	-	~240	273(4.48)	351(4.48)	~390
9	~215	-	~250	270(4.36)	350(4.20)	~400
10	~210	-	~250	~270	349(4.33)	~400
11	203(4.84)	-	254(4.60)	301(4.36)	351(4.39)	~400
12	214(4.54)	-	~235	271(4.33)	350(4.04)	~410
13	~215	-	260(4.37)	~278	350(4.09)	~410
14	213(4.61)	-	~245	271(4.37)	350(4.21)	~400
15	~215	-	~245	288(4.25)	350(4.28)	~400

ture is similar, but the long-wavelength band is almost symmetrical; the inflection is absent. The spectra of the

complexes with compositions donor/acceptor 1:1 and 1:2 do not differ considerably.

The main IR frequencies of the SB are not changed significantly in their PA complexes; the frequencies of the =NH-group are not detectable, and thus a PT from the OH group of PA to the nitrogen atom of the azomethine group can be excluded. The γCH bands of the donors shift to higher wavenumbers, indicating a decrease in the π-electron density as a consequence of the intermolecular CT. The $\nu_s NO_2$ band of the acceptor shifts to lower wavenumbers (from 1352 to 1325-1340 cm^{-1}); the increased π-electron density favours a higher polarization of the nitro group, and accordingly a lower N=O bond order.

We presume that, in the spectra of methanolic solutions of 2-7 and 2a-7a, the range 210-320 nm contains the bands of the π* ← π transitions of both the donor and acceptor molecules, while the 350 nm band involves the band of PA itself (λ_{max} = 350 nm, log ε = 4.02), the band of the intermolecular CT, and the band (~ 420 nm) of the tautomeric equilibrium system characteristic of SB of this type [9]. It seems that the interaction with PA does not prevent the formation of the hydrogen-bonds within the SB molecule. In contrast with the example described above, the spectra of 1 and 1a are different from those of the other compounds discussed, whereas those of the parent SB and of 1 and 1a are similar (Fig. 2), and do not change in different solvents.

It is interesting that, while the parent SB undergoes hydrolysis relatively rapidly in concentrated sulphuric acid solution, the PA complexes remain almost unchanged during 60-80 min (Fig. 1/3).

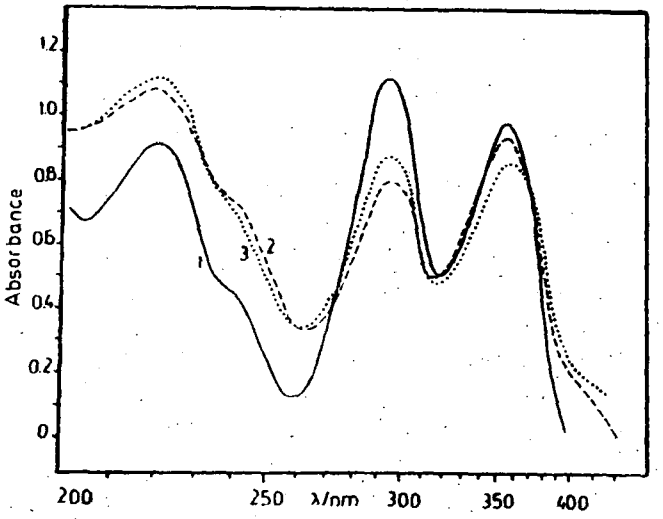


Figure 2.: Spectra of methanolic solutions of the SB(n=0) (1), $c=4.2.10^{-4}$; 1a (2), $c=4.3.10^{-4}$; and the 1:1 DPA complex (3), $c=2.3.10^{-4}$ mol/dm^3; d=0.1 cm.

B) *PA complexes 8-15*. The spectral structures of the PA complexes (Table III) do not differ fundamentally from that of the corresponding SB [14]; the characteristic 270 nm band is unambiguously observable. In the spectra of the SB, there is a flat part between 280 and 350 nm, with an indistinct band at around 340 nm [14], whereas the spectra of the PA complexes (Figs 3 and 4) display a high-intensity band in this region. The calculated curve and the measured one exhibit a considerable intensity difference, from which we conclude that this range contains bands of different origins, as discussed above. The high-intensity bands below 300 nm result from the $\pi^* \rightarrow \pi$ transitions of the aromatic systems. It is re-

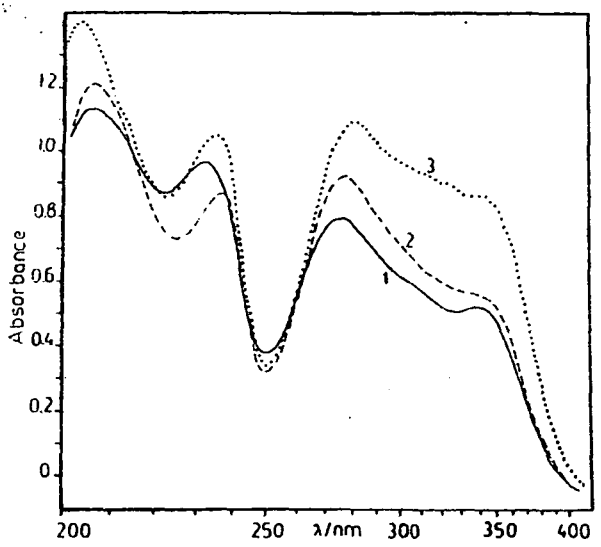


Figure 3.: Spectra of methanolic solutions of 14 (1), $c=3.8.10^{-4}$; 9 (2), $c=6.23.10^{-4}$; and 15 (3), $c=5.09.10^{-4}$ mol/dm^3; d=0.1 cm.

markable that the -NH-R heteroaromatic ring scarcely influences the spectra; the complexes of 8 and 9, for example, even when R=H, give totally similar spectra.

In the IR spectra, a broad, medium-intensity band exists, at around 3000-3100 cm^{-1}, due to the νOH vibration. The strong bands between 1630 and 1660 cm^{-1} correspond to the stretching vibration of the C=N bonds, while there is a strong, broad band between 1550 and 1570 cm^{-1}, which can be assigned as a $\nu_{as}NO_2$ vibration. If there is some other, for example n-π interaction, two $\nu_{as}NO_2$ bands appear; one of these is again equal to or greater than the frequency of the

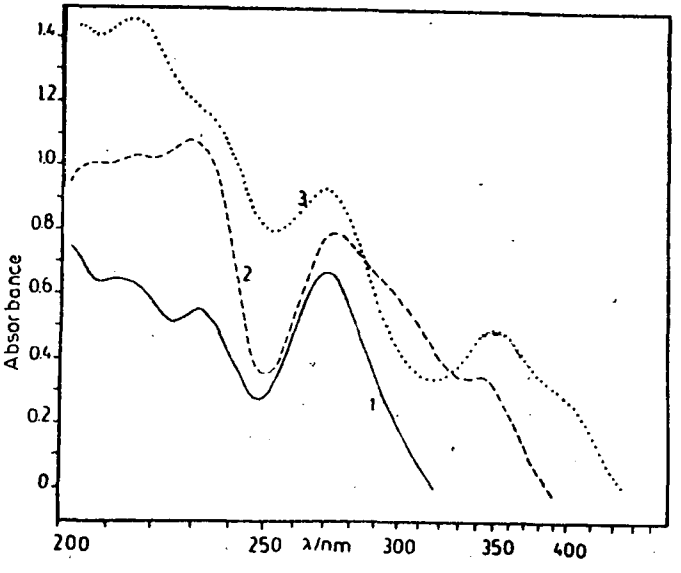


Figure 4.: Spectra of methanolic solutions of the parent sulphonamide of 12 (1), $c=3.8.10^{-4}$; the SB (2), $c=3.9.10^{-4}$; and 12 (3), $c=4.4.10^{-4}$ mol/dm^3; d=0.1 cm.

single band of PA, while the second band is located at lower frequencies [16]. The bands of the $\nu_{as}SO_2$ and ν_sSO_2 vibrations are also well distinguishable in the range of 1340-1370 and 1150-1170 cm^{-1}, respectively. It can be stated that the IR spectra show differences only in the range of the skeletal vibrations; the other ranges are similar. Unfortunately, the spectra are extremely rich in bands, so a full assignment is difficult.

C) *DPA complexes.* It has long been known that DPA yields slightly soluble compounds with, for example, quaternary ammonium compounds [17] or different organic bases [e.g. 18, 19], but very few data can be found in the literature on the spectral behaviour of molecular complexes of aromatic SB and DPA.

While the molecular complexes of SB with PA are generally yellow or orange, the complexes of DPA are brick-red, scarlet or violet. From the structure of DPA, it is obvious that donor ↔ acceptor PT is impossible, and thus only CT processes need be taken into account.

Salicylidene-polymethylenediamines (1-7) form red crystalline complexes with compositions 1:1 and 1:2, the spectra of which are very similar to those of the PA complexes (Figs 1/4 and 2/3); the long-wavelength band shifts to approx. 410-420 nm, due to the increased conjugation systems. On the basis of the high similarity of the spectra, it is reasonable to assume that the PA and DPA complexes have similar structures, and similar excitation processes play important roles in their spectra.

The salicylidene-alkylamines (16-27) also form molecular complexes with DPA in chloroform or methanolic solution, with composition 1:1. In this series, all the spectra display a high similarity to each other; for a typical example, see Fig. 5. The spectra of these compounds are complicated superpositions of the spectra of the components: the long-wavelength band also contains the band assigned to the CT processes, in which the aldehyde ring of the SB obviously takes part in the interaction.

Molecular complexes 28-30 exhibit different spectral structures. 28 shows two bands (295-310 and 380-430 nm); the latter one has the higher intensity. In the case of 29,

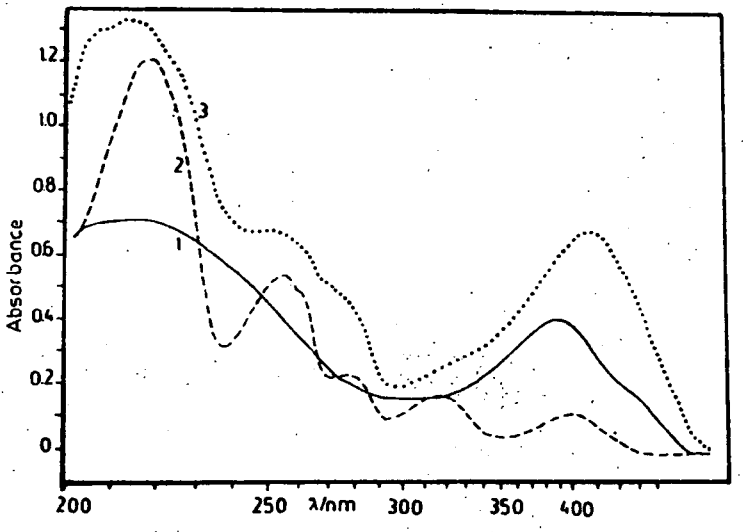


Figure 5.: UV and visible spectra measured in methanol (1) DPA, $c=3.19.10^{-4}$; (2) SB of 26, $c=2.30.10^{-4}$; (3) SB+DPA, $c_{SB}=1.9.10^{-4}$, $c_{DPA}=2.15.10^{-4}$ mol/dm^3; d=0.1 cm.

the bands lie closer together (340-360 and 410-430 nm) and they have comparable extinction coefficients, while in the spectra of 30, only one, high-intensity, broad band exists (Table IV, Fig 6).

The visible band is in every case broad and asymmetric. Accordingly, we presume that this band contains not only the 385 nm band of DPA, but also the band which corresponds to the π - π CT. An important solvent effect is observable in the spectra of 29-34 (see Tables IV and V), but no relationship can be found between the spectral changes and the

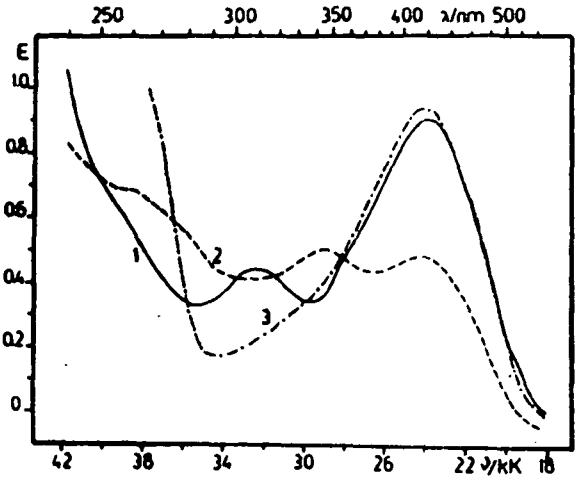


Figure 6.: Spectra measured in methanol. (1) 28, $c=1.11.10^{-4}$; (2) 29, $c=1.30.10^{-4}$; (3) 30, $c=1.30.10^{-4}$ mol/dm^3; d=0.1 cm.

solvent characteristics. The solution spectra do not rigorously obey the Beer law, so we assume that an equilibrium system is formed in solution. In the reflection spectra of 28-30, no band appears in the visible; only a well-defined inflection is observed at around 400 nm.

Selected ^{1}H NMR and IR data on 28-30 are presented in Table IV; partial spectra are shown in Fig. 7. A comparison between the NMR spectra of the studied complexes and those of the components reveals that the signals of the donors and the acceptor are generally shifted to higher and lower δ(ppm) values, respectively. The shifts of the signals due to the pyridine protons are higher, which supports the as-

Table IV

UV, visible (λ_{max}/nm and logε), ^{1}H NMR and IR (cm^{-1}) spectral data on complexes 28-30

Solv.	28		29		30
MeOH	310(4.62)	421(4.92)	348(4.36)	417(4.34)	419(4.86)
$CHCl_3$	~280	~460	349[a]	~400	415[a]
CH_3CN	298(4.43)	423(4.84)	354(4.44)	420(4.43)	438(4.82)
DMSO	309(4.70)	430(4.93)	340(4.32)	434(4.32)	438(4.82)
Acid	303(4.65)	380(4.58)	336[a]	~280	380[a]
Base	300(4.47)	422(4.85)	283(4.42)	406(4.67)	420(4.75)

Assignments			
δCHN	8.80	8.80	8.80
δCH(py-ring)	7.90[b]	7.68[b]	8.08[b]
δCH(ald-ring)	6.95[b]	7.06[b]	6.86[b]
νNH	3100m	3090m	3160m
νC=N	1668s	1625s	1650s
$\nu_{as}NO_2$	1588s	1560s	1585s
$\nu_{s}NO_2$	1285s	1305s[c]	1305s[c]
γC-N(H)	1166m	1170m	1160m
γ_{CH}	908m 767m 738m 718m	908m 764m 740m 718m	907m 765m 738m 720m

a) very poorly soluble; b) the values denote the main frequencies of the multiplets; c) broad complex band

Table V

Spectral data on molecular complexes 31-34

No.	Solv.	λ/nm and logε		
31	MeOH	315(4.46)	-	422(4.74)
	$CHCl_3$	~280	389(4.42)	~440
	CH_3CN	304(4.32)	-	426(4.72)
32	MeOH	318(4.41)	-	420(4.73)
	$CHCl_3$	~280	385(4.33)	~460
	CH_3CN	313(4.34)	-	425(4.79)
33	MeOH	311(4.68)	-	420(5.02)
	$CHCl_3$	~280	380[a]	~470
	CH_3CN	302(4.26)	-	425(4.72)
34	MeOH	261(4.72)	350(4.59)	420(4.58)
	$CHCl_3$	278(4.78)	350(4.66)	~390
	CH_3CN	273(4.69)	347(4.63)	423(4.60)

a) very low solubility

sumption that the pyridine ring is the donating system in the CT interaction. In the case of the parent SB, the two overlapping band systems of the aldehyde and pyridine ring protons lie between 6.7 and 8.1 ppm, while in the spectra of the CT complexes, the two band systems are separated by about 0.8-1.2 ppm.

As a result of the π - π interactions, the characteristic IR frequencies of both the donors and the acceptor change. The sharp νNH frequency of DPA appears at 3095 cm^{-1};

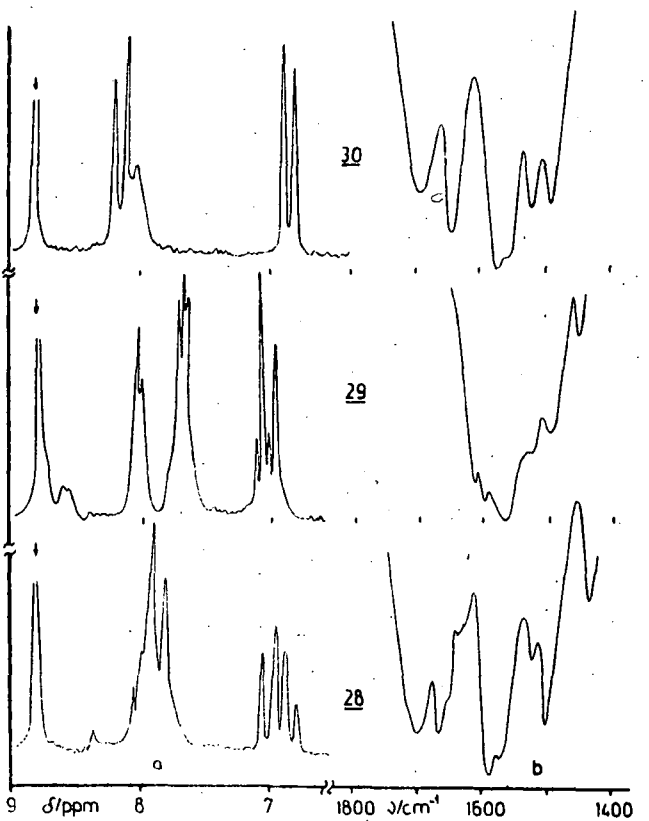


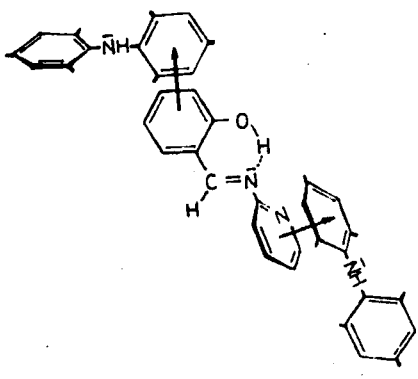
Figure 7.: Partial ^{1}H NMR (δ/ppm) (a) and IR (ν/cm^{-1}) (b) spectra of DPA complexes 28, 29 and 30.

in the spectra of the complexes it is found at around 3090-3200 cm^{-1}. The $\nu_{as}NO_2$ band becomes broader and shows some splitting, indicating a higher differentiation of the

energy states of the nitro groups in the complexes than that in free DPA. A similar change may be observed for the $\nu_s NO_2$ bands. The νC=N band shifts toward higher wavenumbers as compared to the parent SB molecules. In the range 700-910 cm^{-1}, several bands are observed that are due to the γCH vibrations of the different aromatic rings. The γCH bands of the donors shift to higher wavenumbers, which is a criterion for a CT interaction of π - π type [16, 20]. On the other hand, the γCH bands of the pyridine ring exhibit higher shifts in comparison to those of the benzal ring, indicating similarly that the CT takes place between the pyridine ring and the acceptor molecule.

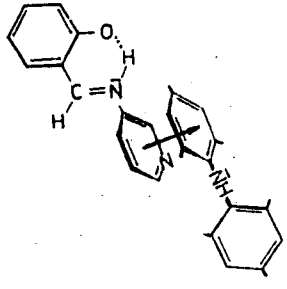
Issa et al. [21] consider that, in the case of benzylydene-aniline derivatives, the aniline ring is the centre primarily contributing to the intermolecular CT interaction; however, in the case of the donor:acceptor 1:2 complexes, the aldehyde ring also takes part in the complex formation. Considering the analytical data and the experimental results, in the case of salicylidene-polymethylenediamines (1-7) and salicylidene-sulphonamide derivatives (8-15), 1:1 + 1:2 and 1:2 complexes are formed, respectively. Since the SB studied are weak electron-donors and PA is a weak acceptor, the resulting CT complex may be expected to be non-ionic. The IR data suggested that the formation of the intramolecular six-membered ring [22, 23] prevents the intermolecular PT; and since the nitrogen lone pair blocks this, the n - π interaction is also improbable; the studied complexes are formed only via an intermolecular CT interaction.

With DPA, 16-27 and 29 form 1:1 complexes, while 28-34 yield 1:2 complexes (not 29). We presume that both the aldehyde and pyridine rings take part in the 1:2 complex formation (Structure V), while in the case of 29 the interac-



V

tion occurs only between the pyridine ring and the aromatic system of the acceptor (Structure VI). However, the dif-



VI

ferent behaviour of 29 is difficult interpret. Saito and Matsunaga [3] have reported amine-PA 1:2 molecular complexes, in which half of the amine molecules act as proton-acceptors, while the other half participate in CT interactions with the picrate ion; i.e. one of the two PA may be a proton-donor, and the other an electron-acceptor. In this case, the IR spectra show the ν-$\overset{\oplus}{N}H_3$ band in the range 2500-300 cm^{-1}. We have no experimental data suggesting the simultaneous operation of CT and PT and/or n - π interactions. It is very difficult to interpret the real structures of these molecular complexes, because strong steric inhibition influences the conformations of both the donor and acceptor molecules.

It is very important to note that it is possible to prepare molecular complexes with different compositions from similar donors and acceptors, depending on the experimental circumstances (temperature, solvent, etc.); this problem requires still further investigations.

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ИЗУЧЕНИЕ СПЕКТРАЛЬНЫХ СВОЙСТВ МОЛЕКУЛЯРНЫХ КОМПЛЕКСОВ ШИФФОВЫХ ОСНОВАНИЙ ОБРАЗОВАННЫХ ИЗ САЛИЦИЛОВОГО АЛЬДЕГИДА И АЛКИЛАМИНОВ, ПОЛИМЕТИЛЕНДИАМИНОВ, СУЛЬФОНАМИДОВ И ПИРИДИНОВ С ПИКРИЛОВОЙ КИСЛОТОЙ И ГЕКСАНИТРОБИФЕНИЛАМИНОМ

И. Часар

Синтетизированы и характеризованы с помощью УФ, видимой, ИК и ^{1}н ЯМР спектроскопии Шиффовые основания (SB) комплексов пикриловой кислоты и гексанитробифениламина, производных салицилового альдегида и алкиламинов, полиметиленодиаминов и аминопиридинов состава **SB.A** и/или **SB.A**$_2$ (где А молекула акцентора). Пикриловая кислота и гексанитробифениламин действуют как акцепторы молекул и связываются к ароматическим кольцам доноров путем межмолекулярного взаимодействия π-π электронного переноса заряда.