



# Characteristic and spectroscopic properties of the Schiff-base model compounds

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## ABSTRACT

Two series of conjugated aromatic imines (Schiff-base model compounds) with different central groups and various side-group substitutions have been synthesized and characterized by elemental analysis, differential scanning calorimetry (DSC) technique, hydrogen nuclear magnetic resonance ( $^1\text{H}$  NMR), Fourier transform infrared (FTIR) and ultra-violet and visible light (UV–vis) spectroscopy measurements. The UV–vis absorption of solutions of these compounds in dimethylacetamid (DMA), chloroform and methanol was investigated in the optical range from 240 to 450 nm, where two distinct absorption bands: at 250–280 and 315–360 nm with the different level of absorption have been observed. The influence of compound molecular structure and polarity of solvent on the absorption spectra and the possible optical transitions have been discussed. Structure of diamines in the azomethine models fundamentally affected their spectroscopic properties and conjugation of  $\pi$ -electrons.

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## 1. Introduction

Azomethines (known as Schiff-bases), having imine groups ( $\text{CH}=\text{N}$ ) and benzene rings in the main chain alternately, and being  $\pi$ -conjugated, exhibit interest as materials for wide spectrum applications, particularly as corrosion inhibitors [1], catalyst carriers [2,3], thermo-stable materials [4–6], a metal ion complexing agents [7] and in biological systems [8,9]. Optical and semiconducting properties of this group of materials have been also widely investigated from many years because of photo- and electroluminescence efficiency with wavelength depended on chemical structure and interesting non-linear optical (NLO) properties of the Schiff-bases in their polymeric form [10,11]. Poly(azomethines) are generally insoluble in common organic solvents, so to evaluate their properties in solutions, analogous model compounds can be used. Synthesis, characterization and spectroscopic properties, particularly substitution effect, tautomerism and influence of polarity and protic of solvents on the UV–visible spectra of solutions of different Schiff-base model compounds and polymers have been described in relatively numerous papers [12–15].

This paper shortly reports synthesis and some typical properties of two series of the Schiff-base model compounds, obtained by the condensation process of benzaldehyde and seven various diamines. The results of elemental analysis, differential scanning calorimetry (DSC) technique, hydrogen nuclear magnetic resonance ( $^1\text{H}$  NMR), Fourier transform infrared (FTIR) spectroscopy and ultra-violet and visible light (UV–vis) absorption have been used to characterize

these groups of model compounds. UV–vis spectroscopy seems to be the simplest and very sensitive method to investigate the optical transitions and electronic structure for organic and non-organic compounds, both in form of solid states (thin films, foils) and as liquids (solutions). Therefore the absorption spectra of all investigated aromatic Schiff-bases are particularly described and discussed in the present work.

The main aim of this study is to report on the results of typical investigations, performed to characterize the properties of some azomethine model compounds with different central group and various side-group substitutions and to present their absorbance spectra in the different solvents in detail. The substitution and solvent effect on the possible electronic transitions and conjugation of  $\pi$ -electrons and the relationships between the chemical structure of compounds and their spectroscopic properties are discussed.

## 2. Experimental

### 2.1. Synthesis of model compounds

Syntheses of the investigated model compounds were based on the condensation reaction of benzaldehyde and aromatic diamine in the presence of p-toluenesulfonic acid. These processes were carried over by stirring a mixture of 3 mmol of proper diamine, 6 mmol of bezaldehyde and 0.06 g of p-toluenesulfonic acid in 10 ml of DMA and heating during 4 h at temperature of 140 °C. After cooling the reaction mixture was poured into 50 ml of ethanol. Resulting precipitate was filtered, washed with ethanol and crystallized from ethanol.

The schematic route of condensation reaction, used in this work, is shown in Fig. 1. Different diamines: 4,4'-diamino-biphenyl,

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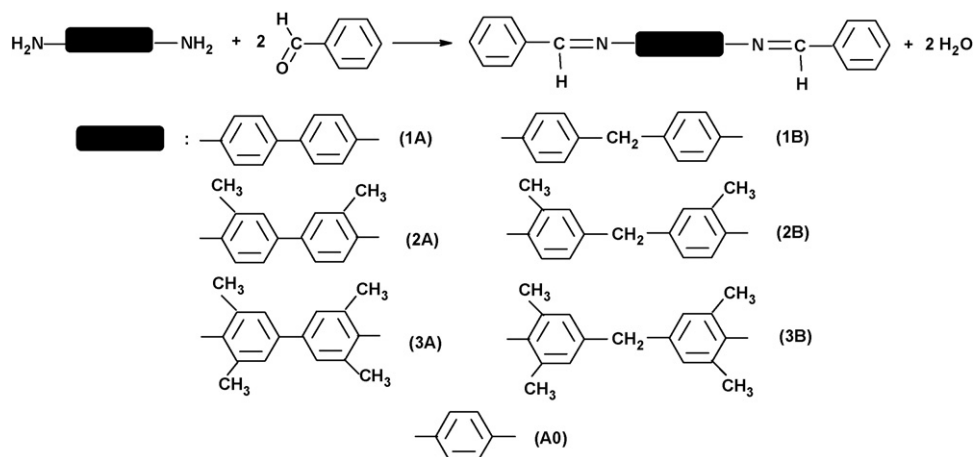


Fig. 1. Synthetic route of the under study azomethines.

3,3'-dimethylbenzidine and 3,3',5,5'-tetramethylbenzidine and also the analogous diamines only with methylene bridge  $\text{CH}_2$  between benzene rings, have been used in this reaction, to synthesize Schiff-base model compounds, called as groups A and B, respectively. Also 1,4-phenylenediamine has been used to obtain the simplest of investigated compounds (named in this text as A0). Chemical structures of all investigated compounds, from both groups, are presented in Fig. 2.

## 2.2. Characterization techniques

Different techniques of the measurements have been used to characterize the Schiff-bases compounds, synthesized by us. The

elemental analysis were performed using a CHNS Perkin Elmer 2400, while the melting points of these compounds were determined by differential scanning calorimetry (DSC) on a TA-DSC 2010 apparatus. The  $^1\text{H}$  NMR spectra were recorded on a Varian Inova 300 spectrometer using chloroform ( $\text{CHCl}_3$ ) as a solvent and tetramethylsilan (TMS) as an internal reference. The infrared spectra were acquired on a DIGITLAB FTS-40A Fourier transform infrared spectrophotometer in the range of  $4000\text{--}400\text{ cm}^{-1}$ , at a resolution of  $2\text{ cm}^{-1}$  and for an accumulated 32 scans and all samples were analyzed in a form of pellets in potassium bromide.

Our measurements of the optical absorption were performed at room temperature on a BECKMAN Acta M-IV (UV-vis-NIR) two beams spectrophotometer, where a deuterium lamp is a source

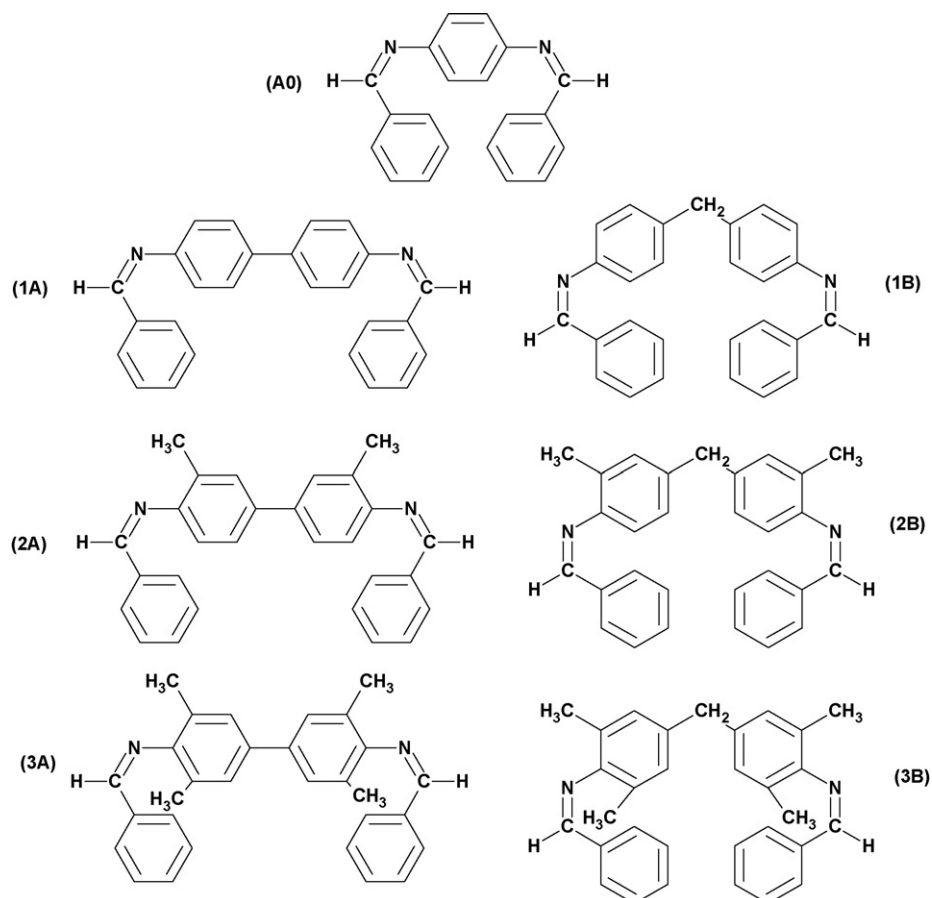


Fig. 2. Chemical structures of the azomethine model compounds.

of ultraviolet (200–380 nm) and a tungsten lamps are used for visible (380–800 nm) and near infrared (800–3300 nm) parts of light. The obtained Schiff-base model compounds are well soluble in the majority of common solvents. Therefore to investigate optical properties of these compounds we have used three different solvents: the most polarity and aprotic dimethylacetamid (DMA)  $\text{CH}_3\text{CON}(\text{CH}_3)_2$ , the polarity and protic methanol  $\text{CH}_3\text{OH}$  and the non-polar three chloromethan (chloroform)  $\text{CHCl}_3$ . The dipole moments ( $M_n$ ) in debyes (D) of these solvents are: 3.92 D for DMA and 1.60 and 1.01 D for methanol and chloroform, respectively. Absorption spectra of investigated azomethine solutions were recorded, using the quartz cells, within the spectral range from 240 to 450 nm, where absorption bands have been observed. The concentration of these solutions has been on the level  $10^{-5}$  mol/l, suitable for the UV–vis optical investigations.

### 3. Results and discussion

Some obtained parameters, of the investigated aromatic Schiff bases, as the results of elemental analysis,  $^1\text{H}$  NMR and FT-IR investigations and the temperature of melting points, are gathered in Table 1.

#### 3.1. Elemental analysis and thermal properties

The results of the elemental analyses, of these two groups of azomethine compounds, are collected in Table 1 and compared with the calculated values of carbon C, hydrogen H and nitrogen N contents. This comparison, shows rather good agreement between calculated and found values and some deficiency of carbon content may be a result of the difficulties in burning these high-temperature compounds.

Melting points of the investigated model compounds strongly depend on their structure. Generally, relation between the structure and melting points exhibit the same direction for both series where the presence of substituents is taken into consideration. Models 1A and 1B, from unsubstituted diamines, melt at higher temperature, than their analogs with one methyl group at ortho-position in each aromatic ring (2A, 2B), that is a common rule. However, the presence of two methyl groups at ortho-position to amine nitrogen in each ring causes increase melting points of the models (3A, 3B) in comparison to these with one methyl group (2A, 2B). Similar behavior was observed for polyketaniles [11,16] and polyimides [17–19]. It seems to indicate that two methyl groups at ortho-position make the molecule more rigid. Rigidity is also the reason why presence of two aromatic rings increases the melting points of the model 1A in comparison with model A0. On the other hand  $-\text{CH}_2-$  group between aromatic ring in diamine (group B) lowers the melting point of these models, due to decrease of rigidity, as comparison with the analogs with direct connected rings (group A).

#### 3.2. $^1\text{H}$ NMR spectroscopy and infrared investigation

In the  $^1\text{H}$  NMR spectra of investigated compounds, singlet signal due to hydrogen atom present in imine group and multiplex confirming protons in aromatic rings were recognized. The most interesting fact is a shift of imine group signal position, depending on the model structure, as it can be seen in Table 1. In the models from unsubstituted diamines (1A,1B) imine proton signal is shifted towards lower field in comparison with their substituted analogs. For the both series, the presence of methyl substituents causes the shift of this signal to higher field. Shift of the imine proton signal towards higher field, indicates the worse conjugation of imine group  $\pi$  electrons with phenyl ring  $\pi$  electrons. Introduction the  $-\text{CH}_2-$  bridge between two aromatic rings (group B) influences on the shift of imine proton signal towards higher fields as compare to the models with biphenyl type structure (group A).

To analyze the infrared spectra of investigated azomethine compounds the position of  $\text{C}=\text{N}$  bond is considered (Table 1). From the literature data [20,21] follows that free, isolated  $\text{C}=\text{N}$  groups absorb at about  $1660\text{ cm}^{-1}$  and this band is associated with the stretching vibrations of this group. The frequency of this band strongly falls with conjugation of the  $\text{C}=\text{N}$  group with phenyl ring due to diminishing at the energy of the  $\text{C}=\text{N}$  bonds and delocalization of nitrogen lone pair into iminic double bond.

As it is seen from Table 1, for the simplest of investigated azomethine (A0), with one phenyl ring between nitrogen atoms, the band characteristic for stretching vibration of the  $\text{C}=\text{N}$  group appears at  $1617\text{ cm}^{-1}$ . Introduction the additional phenyl ring between the nitrogen atoms (1A) causes that this band shifts to  $1622\text{ cm}^{-1}$ . It indicates that conjugation of the  $\text{C}=\text{N}$  bonds and phenyl ring was decreased. Separation of these phenyl rings by the  $\text{CH}_2$  group (1B) gives the frequency of analyzed band rise to  $1625\text{ cm}^{-1}$ , which proves that the further diminishing at conjugation takes place.

Relatively, the higher influence on the position of this band is observed after substitution the phenyl ring connected with nitrogen atom by the methyl groups. Generally, the presence of substituents causes the shift of band in the direction to higher wavenumbers, being more significant for two ortho-methyl groups in each imine ring. In the case of substituted of four methyl groups (3A) the shift is equal to  $17\text{ cm}^{-1}$  in comparison to its unsubstituted model compound (1A). It can be noticed that for azomethine with two phenyl rings substituted by methyl group in ortho-position (2A) only  $6\text{ cm}^{-1}$  shift is recorded in relation to unsubstituted model (1A). Thus it can be proved that the effect of acting of four methyl groups is more than twice higher than that of two methyl groups substituting phenyl rings in ortho-positions.

Analyzing the positions of this band for the compounds from the group B (with  $-\text{CH}_2-$  bridge connecting the phenyl rings) we can notice that the shifts of this band are smaller than ascribed above. In the case of azomethine with two phenyl rings substituted by two methyl groups in ortho-positions (2B) this shift is equal to  $5\text{ cm}^{-1}$ , while for the azomethine with two phenyl rings

**Table 1**  
Some properties of investigated azomethine compounds.

	Elementary analysis–anal. [% $\pm 0.1\%$ ] (calculated) [%]			Melting point $T$ [ $^{\circ}\text{C}$ ]	$^1\text{H}$ NMR $\delta$ [ppm $\pm 0.01$ ]	FTIR $\lambda$ [ $\text{cm}^{-1} \pm 1\text{ cm}^{-1}$ ], –HC=N–(KBr)
	C	H	N			
A0	84.50 (84.57)	5.37 (5.59)	9.73 (9.84)	138	8.51	1617
1A	86.29 (86.63)	5.48 (5.60)	7.67 (7.77)	239	8.57	1622
2A	86.52 (86.55)	6.13 (6.24)	7.30 (7.21)	154	8.42	1628
3A	85.96 (86.48)	6.77 (6.79)	6.76 (6.73)	169	8.27	1639
1B	83.35 (84.76)	8.96 (7.92)	7.30 (7.32)	151	8.44	1625
2B	86.04 (86.52)	6.60 (6.52)	6.86 (6.96)	94	8.37	1630
3B	86.06 (86.45)	7.32 (7.04)	6.33 (6.51)	112	8.21	1638

substituted by four methyl groups at 2,6 and 2'6' positions (3B)  $13\text{ cm}^{-1}$  shift is observed in relation to azomethine with unsubstituted rings (1B). It seems to be interesting that almost the same positions of these bands ( $1628$  and  $1630\text{ cm}^{-1}$ ) is recorded for azomethines with two methyl groups (2A and 2B, respectively) and also in the case of azomethines with four methyl groups the position of this band is similar  $1639\text{ cm}^{-1}$  for 3A and  $1638\text{ cm}^{-1}$  for 3B. It can be concluded that differences in FTIR spectra of the Schiff-base model compounds, described above, are results of changing in charge distribution in the compound chains after substitution between nitrogen atoms.

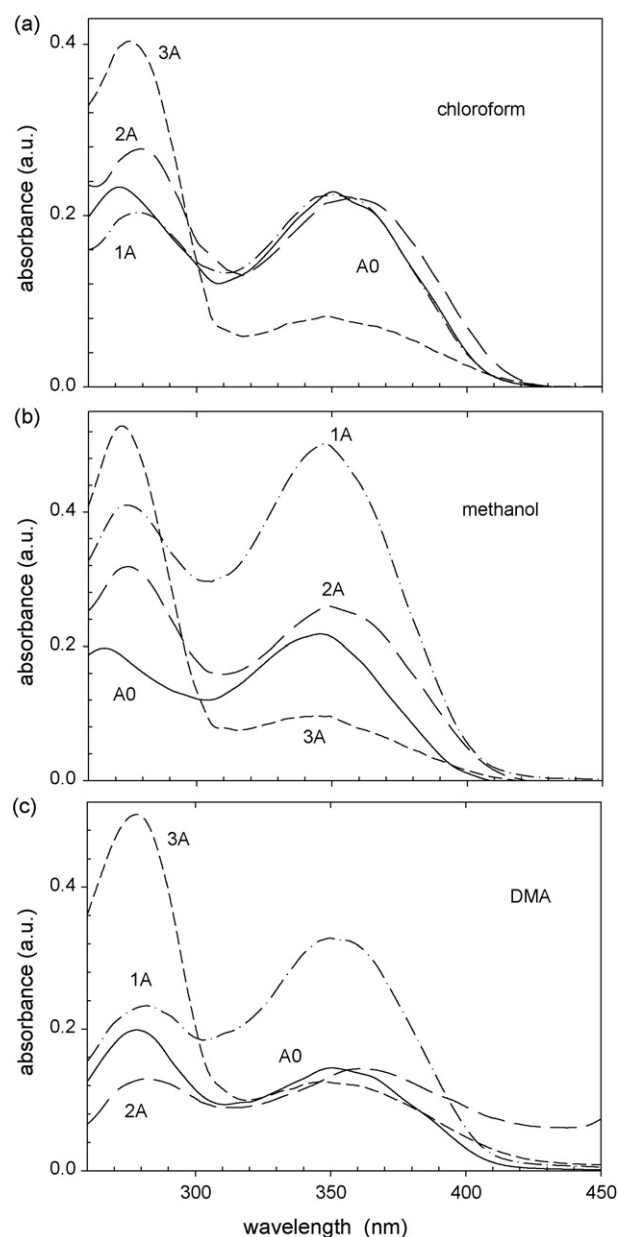
### 3.3. UV-vis absorption spectra

Absorbance spectra, within the optical range from 240 (or 260) to 450 nm, of the solutions in chloroform, methanol and DMA for groups A and B of the Schiff-base model compounds, obtained by us, are seen in Figs. 3(a–c) and 4(a–c) suitably. The short-wave's border of UV spectrum of solution is limited by a permeability of solvent, i.e. 240 nm for chloroform and methanol, 260 nm for DMA. All investigated solutions had the same concentration  $10^{-5}\text{ mol/l}$ . Two absorbance bands at  $\lambda_1$  (250–280) nm and at  $\lambda_2$  (320–380) nm are distinctly presented, in all these spectra. Both, position and intensity of absorption bands depend mainly on the chemical structure of compound but also the polarity of solvent influences these spectra. The long-wavelength absorption bands, seen at  $\lambda_2$ , are due to interband transitions between delocalized ( $D \rightarrow D^*$ ) states which can be derived from interaction of benzene and azomethine dimer  $\pi$ -orbitals, while absorption bands at short-wavelength range ( $\lambda_1$ ) are attributed to interband localized (L) to delocalized ( $D^*$ ) and vice versa, i.e. ( $D$  to  $L^*$ ) transitions. The intensity of absorption bands is usually defined by an extinction coefficient ( $\varepsilon$ ) where  $\varepsilon = A/(c \cdot l)$ ,  $A$  is the absorbance,  $c$  is the concentration of solution,  $l$  is the length of optical way (for instance: the length of quartz cell) but also may be characterized as the value of absorbance ( $A$ ) when concentration of all investigated solutions and conditions of measurements are the same.

The coordinates of absorption bands maximums, i.e. the wavelengths  $\lambda_1$ ,  $\lambda_2$  and the values of absorbance  $A_1$ ,  $A_2$  and also the ratios ( $A_1/A_2$ ) for all investigated azomethine solutions in DMA, chloroform and methanol are gathered in Table 2, in turn.

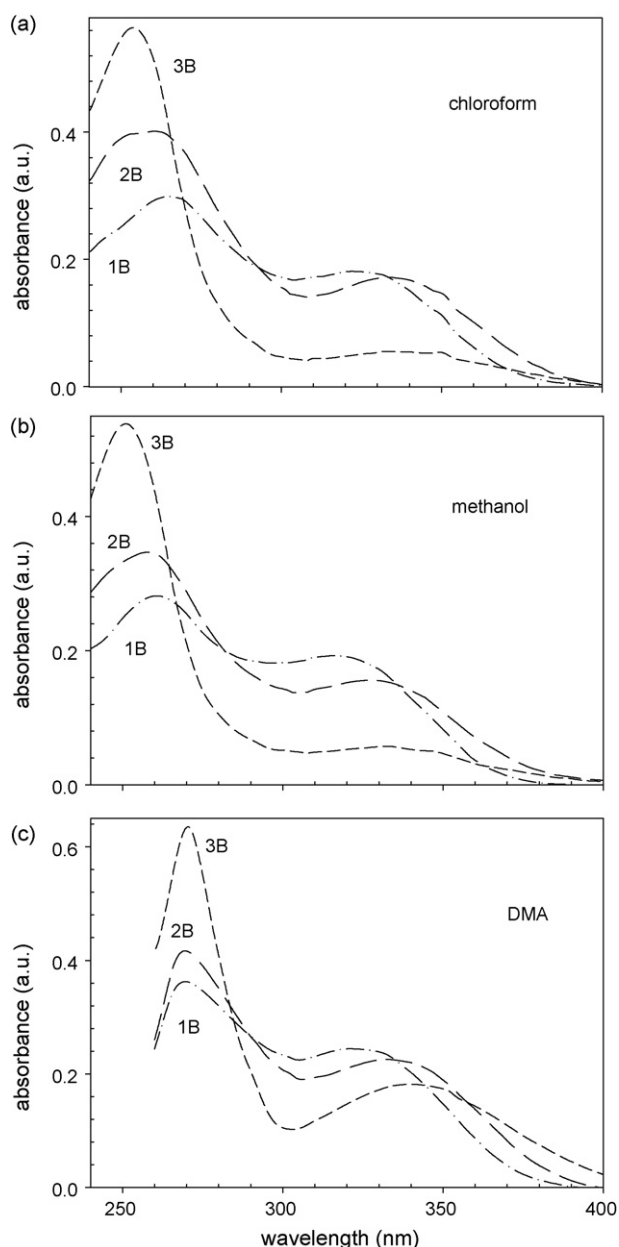
#### 3.3.1. Influence of chemical structure

**3.3.1.1. Effect of  $\text{CH}_3$  side-group substitutions.** As it is seen in Figs. 3 and 4, the clearest feature of spectra for both series of investigated compounds, for all solvents, is the fact that the value of absorbance  $A_2$  decreases and simultaneously  $A_1$  increases with the presence of methyl side-group substitutions in the structure of molecules. This causes that the value of ratio ( $A_1/A_2$ ) distinctly increases (see Table 2) both for compounds from groups A and B. The long-wavelength absorption band at  $\lambda_2$  derives from the interactions of  $\pi$ -orbitals of phenyl and imine group and when the methyl units  $\text{CH}_3$  (one or two) are incorporated to amine ring (di- or tetra-methylbenzidine diamine used to obtain azomethine compound) these interactions are smaller due to the conformation of molecule. As it is known, for bezyldeneaniline twist of the aniline ring out of  $\text{C}=\text{N}=\text{C}$  plane is  $55^\circ$  [22] and similarly, the molecules of our investigated compounds are non-planar. Additionally, as effect of  $\text{CH}_3$  side-group substitutions the conformation increases and delocalization of electrons decreases, what leads to the smaller intensity of absorption band at  $\lambda_2$  (see Figs. 2 and 3). Simultaneously, the role of localized states and the localized  $\rightleftharpoons$  delocalized transitions increases, what causes the increase of the level of the short-wavelength absorption band at  $\lambda_1$ . However, for A0 compound the intensity of the bands are almost equal, i.e. the values of ratio  $A_1/A_2$  are about “1” in all solvents (see Table 2).



**Fig. 3.** Absorption spectra of investigated compounds (group A) as solutions in the different solvents: (a) chloroform, (b) methanol, and (c) DMA; wavelength range 260–450 nm, the same for all solvents.

The influence of the presence of  $\text{CH}_3$  side-groups substitutions on the positions of absorbance maximum  $\lambda_1$  and  $\lambda_2$  is not so clearly. For almost all compounds and solvents, the position of the first maximum  $\lambda_1$  moves insignificantly to the shorter wavelength (hypsochromic effect) with enlargement the chemical structure. For compounds from the group A the wavelength of the second absorbance maximum at  $\lambda_2$  is the longest for one with phenyl rings substituted by two methyl groups in ortho-position (2A) in all solvents. In the case of compounds from group B the value of  $\lambda_2$  moves to the longer wavelength (bathochromic effect) with the increase of amount side-group, except the solutions in chloroform. Simultaneously, the wing of long-wavelength absorption band at  $\lambda_2$  determines the energy gap of compounds. Insignificant changes of its position indicate on the small influence of  $\text{CH}_3$  side-group on the length of conjugated part of compound molecule.



**Fig. 4.** Absorption spectra of investigated compounds (group B) as solutions in the different solvents: (a) chloroform, (b) methanol, and (c) DMA; wavelength range 240–400 nm, the same for all figures.

**3.3.1.2. Effect of presence of  $\text{CH}_2$  bridge.** To estimate the influence of the  $\text{CH}_2$  bridge between phenyl rings (group B) on the results of absorbance for investigated azomethine compounds one can compare the position and value of absorbance maximum, suitably for groups A and B (1A with 1B, 2A with 2B, 3A with 3B in turn, in each solution). Both  $\lambda_1$  and  $\lambda_2$  moves distinctly hipsochromic for compounds from group B in comparison with these for compounds from group A. The presence of  $\text{CH}_2$  unit between phenyl rings causes “break” of the conjugation system and diminishing the length of conjugated part of molecule what leads to the hipsochromic transition of the long-wavelength absorption band.

Characteristic feature of these spectra of compounds from series B, compared to A (except A2 and B2 in methanol) is the increase of absorbance  $A_1$  and simultaneously decrease of  $A_2$ , what indicate that due to the presence of  $\text{CH}_2$  unit between phenyl rings the conformation of molecule increase. As it is described above, the same result, but more clearly, have been observed for  $\text{CH}_3$  side-group

**Table 2**

Absorption bands parameters of investigated compounds solutions in the different solvents.

	$\lambda_1$ [nm]	$\lambda_2$ [nm]	$A_1$	$A_2$	$A_1/A_2$
<b>DMA</b>					
A0	278.35	350.50	0.198	0.145	1.366
1A	281.87	349.87	0.232	0.328	0.709
2A	281.95	361.15	0.129	0.147	0.901
3A	278.20	347.20	0.502	0.125	4.005
1B	269.60	320.90	0.362	0.244	1.485
2B	269.40	332.50	0.333	0.181	1.847
3B	270.44	340.04	0.640	0.081	7.902
<b>Methanol</b>					
A0	265.88	345.75	0.197	0.218	0.904
1A	274.00	347.50	0.410	0.502	0.818
2A	274.62	348.75	0.318	0.260	1.220
3A	272.25	349.13	0.528	0.096	5.485
1B	260.63	316.00	0.282	0.192	1.464
2B	257.75	327.50	0.347	0.156	2.226
3B	251.00	331.87	0.538	0.057	9.376
<b>Chloroform</b>					
A0	271.40	350.46	0.233	0.228	1.022
1A	278.43	346.99	0.203	0.226	0.901
2A	279.48	351.30	0.278	0.222	1.250
3A	275.17	348.46	0.404	0.083	5.540
1B	264.67	322.01	0.299	0.181	1.650
2B	260.37	333.24	0.401	0.172	2.336
3B	253.75	332.71	0.564	0.055	10.255

substitutions. As it is seen in Table 2, the ratio ( $A_1/A_2$ ) increases about two times, for group B, compared to results for analogous compounds from the group A, in all solutions.

### 3.3.2. Influence the polarity of solvent

For all compounds, from both groups of investigated compounds, the wavelength of the first absorbance maximum  $\lambda_1$  moves to the longer values (bathochromically) with increase polarity of solvent, and then the largest value of  $\lambda_1$  is observed for solutions in DMA. The bathochromic shift of the wavelength of second absorbance maximum  $\lambda_2$  is not so evident as in the case of the  $\lambda_1$  value. Only for two compounds from group A and one from group B the largest value of  $\lambda_2$  is for solutions in DMA. In the case of three left compounds the differences between values of  $\lambda_2$  in these three solvents are not so significant. The bathochromic displacement of absorption bands with increase polarity of solvent is usually typical for  $\pi$  types optical transitions. It seems, that polarity of solvent more affect some substituted hydroxy Schiff bases, as it is reported in [13].

Solvents can also influence electronic transitions intensities, what is considered in [23]. In our investigations the values of absorbance maximum  $A_1$  and  $A_2$  seems to be not clearly depended on polarity of solvents. But the ratio  $A_1/A_2$  in the most of cases, both for groups A and B, increases with decrease polarity of solvent (except A0). In the case of chloroform, almost non-polar solvent, the values of  $A_1/A_2$  ratios are the largest, what indicate at the greater role of localized states and transition between localized and delocalized states. Different situation is seen for A0, where the role of localized states is the largest in the most polar of the used solvents, i.e. in DMA.

## 4. Conclusion

The results of our investigations confirmed that structure of the diamine in the azomethine model compounds influenced strongly

on their spectroscopic properties and conjugation of  $\pi$ -electrons. Significant effect is seen when the methyl groups are present at ortho-positions to nitrogen atom in N-phenylene ring. These methyl substitutes causes the shift of imine proton signal towards to higher field at  $^1\text{H}$  NMR spectra and the shift of the  $\text{C}=\text{N}$  bond position to higher wavelength numbers at infrared spectra, in the relation to the spectra for analogous unsubstituted model compounds. The same direction of the peaks shift, but not so significant, have been observed at  $^1\text{H}$  NMR and FTIR spectra, when  $-\text{CH}_2-$  bridge connected two aromatic rings, as compare to the spectra of models with biphenyl type structure. These  $^1\text{H}$  NMR and FTIR results indicates that conjugation between imine group  $\pi$ -electrons and aromatic ring  $\pi$ -electrons and the role of delocalized states decreased for substituted compounds.

On the basis of the UV-vis absorption of azomethine compounds, it is worth to point out, that the methyl side-group substitutions cause some different changes of spectra than the presence of  $\text{CH}_2$  unit inside the molecule structure. Unexpectedly, the ortho-methyl groups in imine rings have not cause the significant shifts of the absorption bands positions. The main effect of the substitution  $\text{CH}_3$  side-groups is the rapid decrease of the long-wavelength absorption band intensity and simultaneously increase the level of the short-wavelength absorption band. The same type of changes of the bands intensity but definitely smaller and additionally clearly hipsochromic transition of the long-wavelength band have been observed for compounds with  $\text{CH}_2$  bridge as compare with the models with biphenyl in their structure. Considering these changes of spectra we can notice that the methyl side-groups substituted to imine ring in ortho-position cause the increase of molecule conformation what leads to the worse conjugation and decrease of the electrons delocalization. The presence of the  $-\text{CH}_2-$  unit between phenyl rings also increases conformation but additionally diminishes the length of conjugated part of molecule.

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