ISSN 1070-3632, Russian Journal of General Chemistry, 2009, Vol. 79, No. 2, pp. 258–263. © Pleiades Publishing, Ltd., 2009.
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Synthesis of *N,N′*-bis-[3-alkoxy-4-(hydroxy, alkoxy, acyloxy)-phenylmethylene- and -Phenylmethyl]-1,3-phenylenediamines

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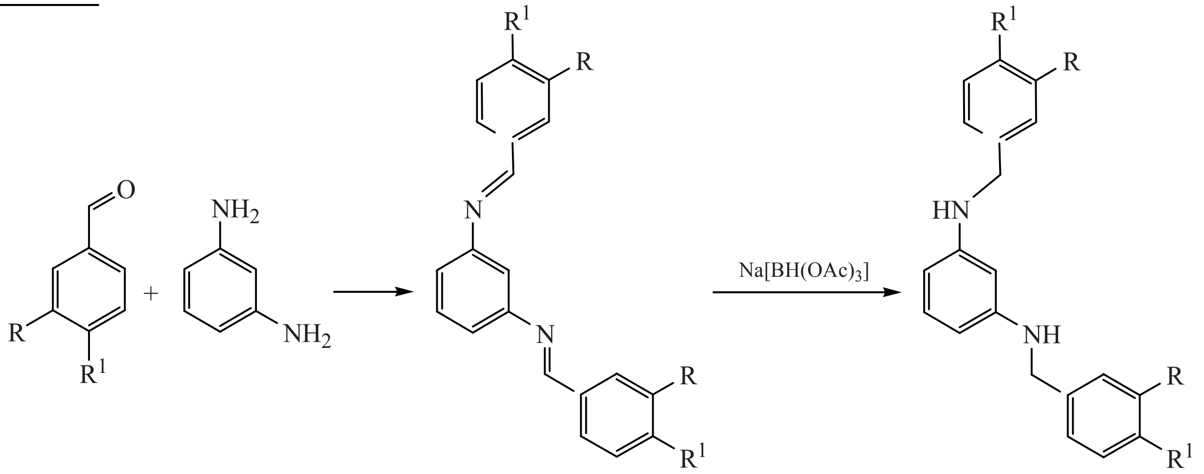
Received September 17, 2008

Abstract—Starting with the vanillin series aldehydes, by reaction with 1,3-phenylenediamine in absolute methanol *E,E-N,N′*-bis-[3-alkoxy-4-(hydroxy-, alkoxy-, acyloxy)phenylme-thylene]-1,3-phenyl-enediamines (Shiff bases) are synthesized from reduction with $Na[BH(OAc)_3]$ in benzene were prepared respective *N,N′*-bis-[3-alkoxy-4-(hydroxy-, alkoxy-, acyloxy)phenylmethyl]-1,3-phenyl-enediamines.

DOI: 10.1134/S1070363209020157

We have reported earlier on the synthesis of *N,N′*-bis-[3-alkoxy-4-(hydroxy-, alkoxy-, acyloxy)phenylmethylene]-1,4-phenylenediamines and 2-[3-alkoxy-4-(hydroxy-, alkoxy-, acyloxy)phenyl]-2,3-dihydro-1*H*-benzimidazoles by the condensation of vanillin series aldehydes with 1,4- and 1,2-phenylenediamines [1, 2]. Aromatic azomethines are interesting substances for producing from tem nano-films and nano-materials and studying their biological activity [3, 4].

The aim of this work is developing a preparative procedure for the synthesis of aromatic bis-azomethines possessing hydroxy, ether and ester groups by condensation of vanillin series aldehydes **I** with 1,3-phenylenediamine **II** (reagents ratio 2:1) in the medium of absolute methanol at the temperature of this solvent boiling. We prepared aromatic bis-azomethines (Shiff bases) (**IIIa–IIIu, IVa–IVk**) with preparative yield 80–90%.



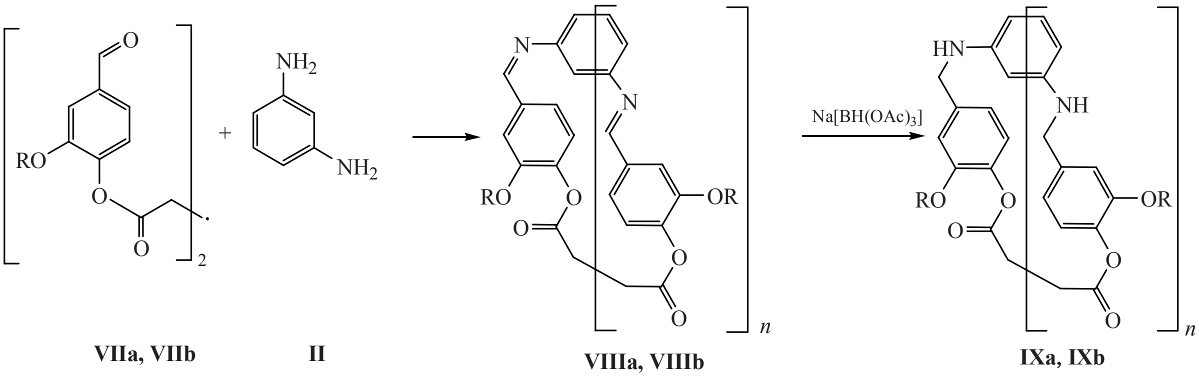
I **II** **IIIa–IIIu, IVa–IVk** **Va–Vu, VIa–VIk**

III, V, $R = R^1 = H$ (**a**); $R = H$, $R^1 = MeO$ (**b**); $R = MeO$, $R^1 = HO$ (**c**), MeO (**d**), MeC(O)O (**e**), EtC(O)O (**f**), PrC(O)O (**g**), $Me_2CHC(O)O$ (**h**), $Me(CH_2)_6C(O)O$ (**i**), $Me(CH_2)_8C(O)O$ (**j**), $Me(CH_2)_{16}C(O)O$ (**k**), $H_2C{=}CMeC(O)O$ (**l**), $C_6H_5CH_2C(O)O$ (**m**), $C_6H_5CHMeCH_2C(O)O$ (**n**), $C_6H_5C(O)O$ (**o**), $4\text{-}ClC_6H_4C(O)O$ (**p**), $2,4\text{-}Cl_2C_6H_3C(O)O$ (**q**), $4\text{-}BrC_6H_4C(O)O$ (**r**), $3\text{-}O_2NC_6H_4C(O)O$ (**s**), MeOC(O)O (**t**), EtOC(O)O (**u**); **IV, VI**, $R = EtO$, $R^1 = HO$ (**a**), MeO (**b**), MeC(O)O (**c**), EtC(O)O (**d**), PrC(O)O (**e**), $Me_2CHC(O)O$ (**f**), $Me_2CHCH_2C(O)O$ (**g**), $4\text{-}MeC_6H_4C(O)O$ (**h**), $3,5\text{-}(O_2N)_2C_6H_3C(O)O$ (**i**), MeOC(O)O (**j**), EtOC(O)O (**k**).

Reaction completed in 10–15 min and proceeded under mild conditions without a catalyst, that promoted retaining of labile ester groups [3].

The obtained bis-azomethines **IIIa–IIIu, IVa–IVk** were reduced with a mild reducing agent sodium tetraacetoxyboron hydride Na[BH(OAc)$_3$] in benzene at 20–23°C. Reduction of aromatic bis-azomethines **IIIa–IIIu, IVa–IVk** to respective secondary diamines **Va–Vu, VIa–VIk** was completing in 18–20 h. Yield of secondary diamines **Va–Vu, VIa–VIk** was practically quantitative, actually 94–96%. Under the conditions of the reduction reaction carried out at specially gathered gentle temperature regime and acid action [5] we did not observe proceeding of side reaction of reduction of ester, nitro and unsaturated methacrylic groups in side chains.

At the condensation of vanillin succinate and vanillal **VIIa, VIIb** with 1,3-phenylenediamine **II** (ratio of the reagents 1:1) by the similar scheme were prepared oligomeric cyclic azomethines (Shiff bases) **VIIIa, VIIIb** in preparative yield 92–93%. Reduction of these compounds with Na[BH(OAc)$_3$] led to oligomeric cyclic amines **IXa, IXb** in 94–96% yield.



VII–IX, R = Me (**a**), Et (**b**), *n* = 1–8.

Structures of the synthesized azomethines and secondary amines **IIIa–IIIu, IVa–IVk, Va–Vu, VIa–VIk, VIIIa, VIIIb, IXa,** and **IXb** are confirmed by the data of elemental analysis, cryoscopic determining of molecular mass (Table 1), IR and ^{1}H NMR spectra. According to the ^{1}H NMR spectra, purity of the obtained compounds is 96±2%.

In the IR spectra of compounds **IIIa–IIIu, IVa–IVk, Va–Vu, Via–VIk, VIIIa, VIIIb, IXa** and **IXb** there are absorption bands of the bonds C–H$_{\mathrm{Ar}}$ in the regions of 3080–3070, 3005–3002, 870–700; C–H$_{\mathrm{Alk}}$ 2970–2830; C=O 1765–1730; C–C$_{\mathrm{Ar}}$ 1610–1590, 1505–1500, 1466–1460, 1418–1410, 1375-1366; C–O 1280–1260, 1210–1190, 1150–1110, 1035–1030 cm^{-1}. In the IR spectra of azomethines **IIIa–IIIu, IVa–IVk, VIIIa,** and **VIIIb** there is a characteristic absorption band of C=N group in the region of 1630–1627 cm^{-1}, that is absent in the spectra of amines **Va–Vu, VIa–VIk, IXa,** and **IXb**. In the IR spectra of amines **Va–Vu, VIa–VIk, IXa,** and **IXb** appears characteristic N–H absorption band in the region of 3420–3375 cm^{-1}. In the IR spectra of nitro compounds **IIIs, IVi, Vs,** and **VIi** occurs absorption bands of NO$_2$ group at 1544–1528 and 1350–1345 cm^{-1}.

In the ^{1}H NMR spectra of compounds **IIIb–IIIu, IVb, Vb–Vu,** and **VIb** the signals of MeO group protons appear as a singlet in the range from 3.85 to 3.92 ppm, in the spectra of compounds **IVa–IVk** and **VIa–VIk** there are the signals of EtO group protons as a triplet at 1.10–1.45 ppm (Me) and a quadruplet at 3.90-4.30 ppm (CH$_2$). The signals of aromatic protons in compounds **IIIa–IIIu, IVa–IVk, Va–Vu,** and **VIa–VIk** are located in the range of 6.90–7.40 ppm. The proton of azomethine group (HC=N) in compounds **IIIa–IIIu** and **IVa–IVk** induces a singlet in the region of 8.45–8.55 ppm, that is characteristic of azomethine *E*-isomers [1–3]. In the ^{1}H NMR spectra of secondary amines **Va–Vu** and **VIa–VIk** there are the signals of CH$_2$ group protons (CH$_2$NH) as a broad singlet in the region of 4.40–4.42 ppm. The ^{1}H NMR spectra of compounds **VIIIa, VIIIb, IXa,** and **IXb** were not recorded due to low solubility of these compounds. By the same reasons their molecular weighs were not determined.

In the IR and ^{1}H NMR spectra of azomethines and secondary amines **IIIa–IIIu, IVa–IVk, Va–Vu, VIa–VIk, VIIIa, VIIIb, IXa,** and **IXb** occur also all absorption bands and proton signals confirming presence of

Table 1. Yields, melting points, data of elemental anlysis and molecular weghts of compounds **IIIa–IIIu, IVa–IVk, Va–Vu, VIa–VIk, VIIIa, VIIIb, IXa,** and **IXb**

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %			*M*	
			C	H	N		C	H	N	found	calculated
IIIa	85	154–155	84.91	5.76	9.35	$C_{20}H_{16}N_2$	84.48	5.67	9.85	275.7	284.4
IIIb	89	113–114	77.03	5.96	7.84	$C_{22}H_{20}N_2O_2$	76.72	5.85	8.13	338.0	344.4
IIIc	80	138–139	70.62	5.43	7.08	$C_{22}H_{20}N_2O_4$	70.20	5.36	7.44	368.2	376.4
IIId	85	122–123	71.48	6.14	6.60	$C_{24}H_{24}N_2O_4$	71.27	5.98	6.93	396.3	404.5
IIIe	90	126–127	68.12	5.36	5.82	$C_{26}H_{24}N_2O_6$	67.82	5.25	6.08	453.8	460.5
IIIf	88	85–86	68.04	5.80	5.43	$C_{28}H_{28}N_2O_6$	68.84	5.78	5.73	481.2	488.5
IIIg	90	138-139	70.03	6.32	5.12	$C_{30}H_{32}N_2O_6$	69.75	6.24	5.42	508.6	516.6
IIIh	87	194–195	69.97	6.34	5.12	$C_{30}H_{32}N_2O_6$	69.75	6.24	5.42	505.9	516.6
IIIi	87	111–112	72.84	7.75	4.05	$C_{38}H_{48}N_2O_6$	72.58	7.69	4.46	620.4	628.8
IIIj	90	92–93	73.90	8.33	3.87	$C_{42}H_{56}N_2O_6$	73.65	8.24	4.09	673.0	684.9
IIIk	82	53–54	76.94	9.92	2.62	$C_{58}H_{88}N_2O_6$	76.61	9.75	3.08	896.5	909.3
IIIl	80	106–107	70.51	5.52	4.14	$C_{30}H_{28}N_2O_6$	70.30	5.51	4.47	504.7	512.6
IIIm	81	137–138	74.69	5.35	4.19	$C_{38}H_{32}N_2O_6$	74.50	5.26	4.57	604.8	612.7
IIIn	84	88–89	75.76	6.14	3.88	$C_{42}H_{40}N_2O_6$	75.43	6.03	4.19	659.8	668.8
IIIo	90	138–139	74.10	4.90	4.36	$C_{36}H_{28}N_2O_6$	73.96	4.83	4.79	576.4	584.6
IIIp[a]	86	96–97	66.39	4.13	4.02	$C_{36}H_{26}Cl_2N_2O_6$	66.16	4.01	4.29	644.7	653.5
IIIq[b]	85	92–93	60.01	3.40	3.72	$C_{36}H_{24}Cl_4N_2O_6$	59.85	3.35	3.88	708.2	722.4
IIIr[c]	86	115–116	58.65	3.72	3.45	$C_{36}H_{26}Br_2N_2O_6$	58.24	3.53	3.77	732.1	742.4
IIIs	88	144–145	64.28	3.95	8.02	$C_{36}H_{26}N_4O_{10}$	64.09	3.88	8.30	665.4	674.6
IIIt	90	120–121	63.87	5.05	5.32	$C_{26}H_{24}N_2O_8$	63.41	4.91	5.69	484.3	492.5
IIIu	85	88–89	65.03	5.58	5.14	$C_{28}H_{28}N_2O_8$	64.61	5.42	5.38	512.3	520.5
IVa	82	56–57	71.56	6.10	6.65	$C_{24}H_{24}N_2O_4$	71.27	5.98	6.93	392.8	404.5
IVb	84	151–152	72.54	6.59	6.22	$C_{26}H_{28}N_2O_4$	72.20	6.52	6.48	418.7	432.5
IVc	88	77–78	69.00	5.87	5.40	$C_{28}H_{28}N_2O_6$	68.84	5.78	5.73	475.0	488.5
IVd	87	82–83	69.98	6.32	5.11	$C_{30}H_{32}N_2O_6$	69.75	6.24	5.42	507.2	516.6
IVe	84	118–119	70.86	6.84	4.82	$C_{32}H_{36}N_2O_6$	70.57	6.66	5.14	536.4	544.6
IVf	88	125–126	70.93	6.79	4.80	$C_{32}H_{36}N_2O_6$	70.57	6.66	5.14	532.5	544.6
IVg	87	143–144	71.62	7.18	4.53	$C_{34}H_{40}N_2O_6$	71.31	7.04	4.89	565.7	572.7
IVh	90	133–134	75.23	5.66	4.09	$C_{40}H_{36}N_2O_6$	74.98	5.66	4.37	651.2	640.7
IVi	88	140–141	75.87	3.65	10.21	$C_{38}H_{28}N_6O_{14}$	75.58	3.56	10.60	781.6	792.7
IVj	82	84–86	64.87	5.62	5.06	$C_{28}H_{28}N_2O_8$	64.61	5.42	5.38	511.6	520.5
IVk	85	132–133	65.84	5.99	4.83	$C_{30}H_{32}N_2O_8$	65.58	5.88	5.11	534.9	548.6

Table 1. (Contd.)

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %			*M*	
			C	H	N		C	H	N	found	calculated
Va	94	120–121	83.49	7.14	9.52	$C_{20}H_{20}N_2$	83.30	6.99	9.71	273.1	288.4
Vb	95	102–103	76.05	7.08	7.86	$C_{22}H_{24}N_2O_2$	75.83	6.94	8.04	340.2	348.4
Vc	95	116–117	69.73	6.52	7.01	$C_{22}H_{24}N_2O_4$	69.46	6.36	7.36	365.0	380.4
Vd	95	109–110	70.86	7.07	6.38	$C_{24}H_{28}N_2O_4$	70.57	6.91	6.86	390.4	408.5
Ve	94	147–148	67.63	6.18	5.74	$C_{26}H_{28}N_2O_6$	67.23	6.08	6.03	451.2	464.5
Vf	95	74–75	68.52	6.62	5.32	$C_{28}H_{32}N_2O_6$	68.28	6.55	5.69	479.5	492.5
Vg	96	121–122	69.45	7.10	5.03	$C_{30}H_{36}N_2O_6$	69.21	6.97	5.38	506.7	520.6
Vh	95	183–184	69.73	7.06	4.97	$C_{30}H_{36}N_2O_6$	69.21	6.97	5.38	508.9	520.6
Vi	95	106–107	72.62	8.35	4.00	$C_{38}H_{52}N_2O_6$	72.12	8.28	4.43	621.0	632.8
Vj	94	88–89	73.51	8.86	3.82	$C_{42}H_{60}N_2O_6$	73.22	8.78	4.07	665.8	688.9
Vk	96	65–66	76.15	10.34	2.90	$C_{58}H_{92}N_2O_6$	76.27	10.15	3.07	901.1	913.4
Vl	94	89–90	69.96	6.33	5.08	$C_{30}H_{32}N_2O_6$	69.75	6.24	5.42	503.4	516.6
Vm	94	124–125	74.42	5.99	4.07	$C_{38}H_{36}N_2O_6$	74.01	5.88	4.54	604.5	616.7
Vn	94	83–84	75.20	6.67	3.88	$C_{42}H_{44}N_2O_6$	74.98	6.59	4.16	660.3	672.8
Vo	96	122–123	73.67	5.62	4.35	$C_{36}H_{32}N_2O_6$	73.45	5.48	4.76	578.8	588.7
Vp[d]	94	78–79	66.06	4.74	3.98	$C_{36}H_{30}Cl_2N_2O_6$	65.76	4.60	4.26	650.0	657.5
Vq[e]	95	87–88	59.88	4.02	3.46	$C_{36}H_{28}Cl_4N_2O_6$	59.52	3.88	3.86	613.4	726.4
Vr[f]	95	106–107	58.21	4.10	3.34	$C_{36}H_{30}Br_2N_2O_6$	57.93	4.05	3.75	732.9	746.5
Vs	94	132–133	64.08	4.53	7.95	$C_{36}H_{30}N_4O_{10}$	63.71	4.46	8.26	660.5	678.7
Vt	94	108–109	63.17	5.76	5.16	$C_{26}H_{28}N_2O_8$	62.90	5.68	5.64	483.6	496.5
Vu	96	96–97	64.34	6.23	5.04	$C_{28}H_{32}N_2O_8$	64.11	6.15	5.34	503.7	524.6
VIa	95	68–69	70.86	6.96	6.53	$C_{24}H_{28}N_2O_4$	70.57	6.91	6.86	393.6	408.5
VIb	94	134–135	71.85	7.39	6.18	$C_{26}H_{32}N_2O_4$	71.53	7.39	6.42	420.6	436.5
VIc	94	71–72	68.63	6.62	5.34	$C_{28}H_{32}N_2O_6$	68.28	6.55	5.69	480.4	492.5
VId	96	69–70	69.62	7.14	5.05	$C_{30}H_{36}N_2O_6$	69.21	6.97	5.38	508.5	520.6
VIe	94	106–107	70.39	7.48	4.75	$C_{32}H_{40}N_2O_6$	70.05	7.35	5.11	539.3	548.7
VIf	95	118–119	70.24	7.39	6.81	$C_{32}H_{40}N_2O_6$	70.05	7.35	5.11	531.5	548.7
VIg	94	134–135	71.03	7.77	4.38	$C_{34}H_{44}N_2O_6$	70.81	7.69	4.86	562.4	576.7
VIh	95	111–112	74.87	6.38	4.03	$C_{40}H_{40}N_2O_6$	74.51	6.25	4.34	632.2	644.8
VIi	94	122–123	57.66	4.23	10.02	$C_{38}H_{32}N_6O_{14}$	57.29	4.05	10.55	778.0	796.7
VIj	94	88–89	64.65	6.28	5.07	$C_{28}H_{32}N_2O_8$	64.11	6.15	5.34	509.8	524.5
VIk	95	117–118	65.45	6.58	4.73	$C_{30}H_{36}N_2O_8$	65.20	6.57	5.07	542.6	552.6
VIIIa	93	>310	68.67	5.05	5.62	$(C_{26}H_{22}N_2O_6)_n$	68.11	4.84	6.11	–	$(458.5)_n$

Table 1. (Contd.)

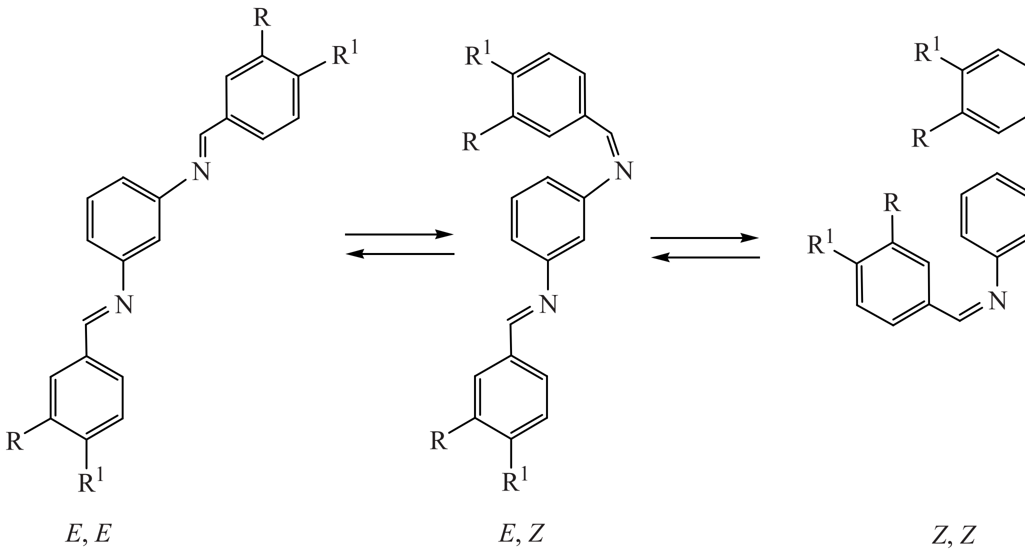
Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %			*M*	
			C	H	N		C	H	N	found	calculated
VIIIb	92	>310	69.70	5.54	5.12	$(C_{28}H_{26}N_2O_6)_n$	69.12	5.39	5.76	–	$(486.5)_n$
IXa	96	>310	68.03	6.91	5.73	$(C_{26}H_{26}N_2O_6)_n$	67.52	5.67	6.06	–	$(462.5)_n$
IXb	94	>310	69.08	6.43	5.30	$(C_{28}H_{30}N_2O_6)_n$	68.56	6.16	5.76	–	$(490.6)_n$

[a] Found Cl, %: 10.31. Calculated Cl, %: 10.85. [b] Found Cl, %: 19.30. Calculated Cl, %: 19.63. [c] Found Br, %: 21.10. Calculated Br, %: 21.53. [d] Found Cl, %: 10.37. Calculated Cl, %: 10.78. [e] Found Cl, %: 19.16. Calculated Cl, %: 19.52. [f] Found Br, %: 21.14. Calculated Br, %: 21.41.

respective structural fragments of ester groups characteristic of this class compounds [1–3].

Some of relatively high melting bis-azomethines (mp above 120°C, see Table 1) showed thermochromic effect owing to reversible transition from *E,E*-configuration of bis-azomethine to less thermodynamically advantageous *E,Z*- and *Z,Z*-confirgurations that occur before the beginning the crystals of melting. This process is accompanied by the change in the form and color of the crystals [1].

For the sudy of the observed thermochromic effect at the melting of the crystals of azomethines we carried out quantum-chemical calculations of the heat of formation (H_f) of *E,E*-, *E,Z*- and *Z,Z*-configuration isomers of bis-azomethines **IIIa, IIId, IIIe,** and **IVd** and obtained from them secondary diamines **Va, Vd, Ve,** and **VId**. The quantum-chemical calculation were carried out in the framework of semiempirical MNDO PM3 approximation [6] with GAMESS program [7]. All bond lengths, bond and dihedral angles of the studied compounds were completely optimized. The following H_f values (kcal mol^{-1}) were obtained: for *E,E*-isomers, 114.9 (**IIIa**), –30.7 (**IIId**), –119.8 (**IIIe**), –138.6 (**IVd**); for *E,Z*-isomers, 115.1 (**IIIa**), –30.1 (**IIId**), –119.1 (**IIIe**), –137.3 (**IVd**); for *Z,Z*-isomers, 115.6 (**IIIa**), –29.0 (**IIId**), –118.6 (**IIIe**), and –136.7 (**IVd**). From these calculations follows that *E,E*-configuration is by 0.2-1.3 kcal mol^{-1} is preferable energetically over *E,Z*- and by 0.7–1.9 kcal mol^{-1} over *Z,Z*-configuration. The calculated H_f (kcal mol^{-1}) values for the respective diamines are: –74.1 (**Va**), –72.1 (**Vd**), –162.7 (**Vd**), and –179.5 (**VId**). Hydrogenation of *E,E*-bis-azomethines to diamines leads to incrase in thermodynamic stability by 40.8–42.9 kcal mol^{-1}. The calculated energy barriers for the interconfigura-tion *E,E*-, *E,Z*- and *Z,Z*-transitions are 8–13 kcal mol^{-1}, that allows to apply the synthesized bis-azomethines as thermo- and light-sensitive molecular nano-switches [8, 9].



EXPERIMENTAL

The IR spectra were recorded on a Fourier transform IR spectrometer Protege-460 of Nicolet, from KBr pellets. The ^{1}H NMR spectra were recorded on a BS-587A (100 MHz) Tesla spectrometer from 5% solutions in $CDCl_3$, chemical shifts were measured from internal TMS. The elemental analysis was carried out on a C, H, N, O, S-analyzer Vario EL-III Elementar, error of determination 0.1%. Molecular weight was determined by the method of cryoscopy in benzene.

Vanillin and vanillal esters **I** were obtained along the procedures in [11–14]. For the syntheses was used 1,3-phenylenediamine **II** of “analytically pure” grade, purity 99%, mp 62–63°C .

***E,E-N,N′*-bis-[3-Alkoxy-4-(hydroxy-, alkoxy-, acyloxy)phenylmethylene]-1,3-phenylenediamines (IIIa–IIIu, IVa–IVk)** *(general procedure)*. A solution of 10 mmol of a vanillin series aldehyde **I** and 5 mmol of 1,3-phenylenediamine **II** in 50 ml of absolute methanol was refluxed for 10–15 min. The hot solution was filtered through a folded paper, cooled and left for 10–15 h at 5°C . The azomethine formed **IIIa–IIIu, IVa–IVk** was separated by filtration on a porous glass filter, washed with a small amount of methanol and dried in air.

***N,N′*-bis-[3-Alkoxy-4-(hydroxy-, alkoxy-, acyloxy)phenylmethyl]-1,3-phenylenediamines (Va–Vu, VIa-VIk)** *(general procedure).* A solution of 5 mmol of bis-azomethine **IIIa–IIIu , IVa–IVk**, 20 mmol of $NaBH_4$, 60 mmol of glacial acetic acid in 70 ml of absolute benzene was left for 18–20 h at 20–23°C . The solution formed was washed with water and 5% aqueous $NaHCO_3$ and solvent was removed in a vacuum. The residue was purified by recrystallization from benzene – hexane mixture.

***E,E*-poly-[3-Alkoxy-4-succinyloxyphenylmethylene]-1,3-phenylenediamines (VIIIa, VIIIb).** A solution of 5 mmol of vanillin or vanillal succinate **VIIa, VIIb** and 5 mmol of 1,3-phenylenediamine **II** in 50 ml of 1:1 mixture of absolute methanol and chloroform was refluxed for 30 min. The hot solution was cooled and azomethine **VIIIa, VIIIb** was separated by filtration on porous glass filter, washed with small amount of methanol and dried in air.

Poly-[3-Alkoxy-4-succinyloxyphenylmethyl]-1,3-phenylenediamines (IXa, IXb). A suspension of 5 mmol of polyazomethine **VIIIa, VIIIb**, 20 mmol of $NaBH_4$, 60 mmol of glacial acetic acid in 70 ml of absolute benzene was left at 20–23°C for 5 days with periodical stirring with magnetic stirrer. The polyamines formed **IXa, IXb** were separated by filtration on a porous glass filter, carefully washed with water, 5% aqueous $NaHCO_3$, again with water and with some amount of methanol, and dried in air.

ACKNOWLEDGMENTS

This work was financially supported by the Foundation for Bsic Research of Republic Belarus (grant X 08-227).

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