Synthesis of 1-(α-Aminobenzyl)-2-naphthol Derivatives, their Structure in Crystal and in Solution

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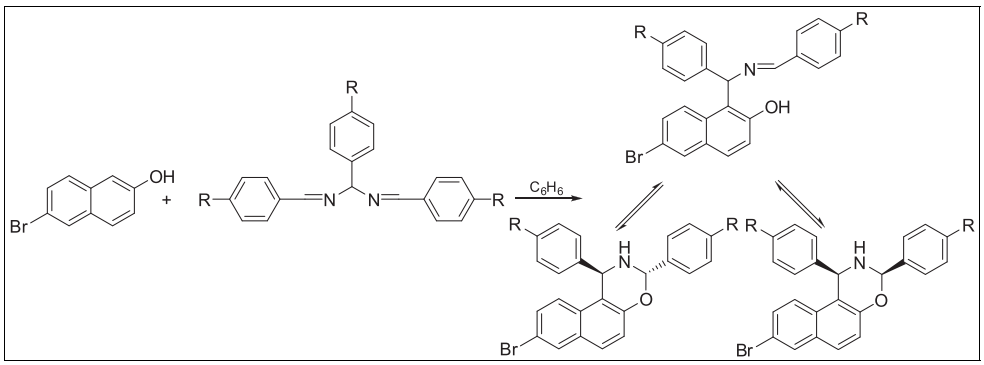
Received December 10, 2012
DOI 10.1002/jhet.1978
Published online 00 Month 2014 in Wiley Online Library (wileyonlinelibrary.com).



The series of tautomeric 1,3-diarylnaphthoxazines (the Betti base precursors) was obtained by the interaction of 2-naphthols and 1,3,5-trisaryl-2,4-diazapenta-1,4-dienes. Their structure has been established in solid state and solution.

J. Heterocyclic Chem., **00,** 00 (2014).

INTRODUCTION

1-(α-Aminobenzyl)-2-naphthol is the chiral compound for the first time obtained by the Italian scientist Mario Betti in the first decade of the last century. Its derivatives have recently attracted the attention of researchers who work on asymmetric syntheses because of the need for new easily accessible chiral enantiopure compounds that can be used as auxiliaries or precursors. 1-(α-Aminobenzyl)-2-naphthols, named in honor of the author of these compounds, the Betti bases, are quite suitable for these purposes because of the ease of synthesis, the availability of the initial reagents, and potential for enantiomeric separation. Surprisingly enough for a long time, this class of compounds did not attract proper attention until recently, when several publications appeared in this field [1,2] owing to the progress in stereochemistry and the synthesis of enantiopure medicines.

RESULTS AND DISCUSSION

Recently, we reported a new approach to the synthesis of 1,3-diaryl-2,3-dihydro-1*H*-naphtho[1,2-e][1,3]oxazines – Betti base derivatives. The method involves the interaction of 2-naphthol **1** with very accessible 1,3,5-trisaryl-2,4-diazapenta-1,4-dienes **2** in molar ratio 3:2 in boiling benzene (Scheme 1) [3]. To expand the boundaries of proposed method, 6-bromo-2-naphthol was used in the reaction with 1,3,5-trisaryl-2,4-diazapenta-1,4-dienes **2c–2f** resulting in formation of **3g–3j** with good yield and purity. In accordance with the literature data [4–8], compounds **3a–3j** in $CDCl_3$ solution are present as a mixture of tautomers **A**, **B**, and **C** because of the ring-chain tautomerism.

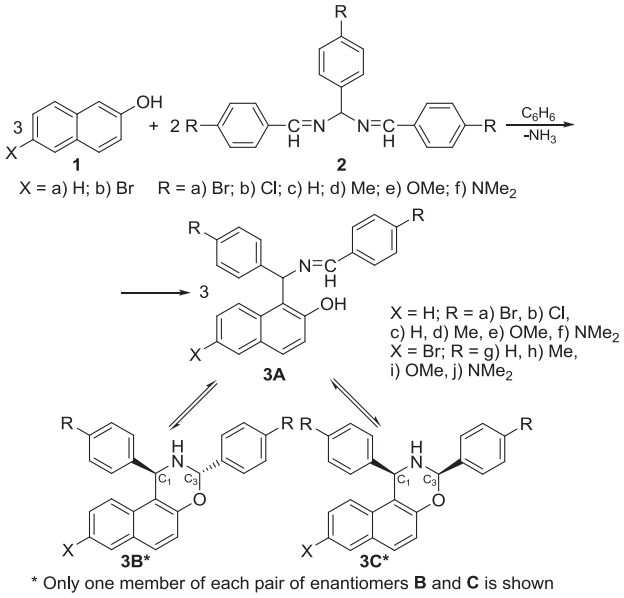
Compounds **3g**, **3i**, and **3j** were previously synthesized using standard Betti method [9,10], the obtained results raising some doubts. The difference in melting points between our and literature data reaches in certain cases about 70°C, and ^{1}H NMR studies exhibit the presence of only one tautomer, which is impossible for this sort of compounds.

Because of significant discrepancies between our and literature data, it is necessary to give full description of obtained products **3g–3j**. As it was expected, all compounds **3g–3j** in the $CDCl_3$ solution exist as a mixture of tautomers **A**, **B**, and **C** because of the ring-chain tautomerism. The ratio of tautomers, as determined by integration of proton signals at C1 and C3, is given in Table 1.

Differentiation and classification of tautomers were carried out in accordance with the literature data [4,5]. It is necessary to note that tautomers' ratio for compounds **3g–3j** is almost the same as tautomers' ratio for 1,3-naphthoxazines derived from 2-naphthol with the same substitutes. Notice that the ratio of imine tautomer increases with rising of donor ability of substituents in aryl fragments and reveals maximum for dimethylamino-substituted derivative.

Thus, condensation products of Betti bases with various aldehydes in solution exist in three tautomeric forms, because of oxazine-imine tautomeric equilibrium. However, these compounds are not as well studied in the solid phase. To

Scheme 1. Preparation of 1,3-diarylnaphthoxazines **3a–3j**.



Scheme 2. Preparation of 1-phenyl-3-arylnaphthoxazines **4a–4d**.

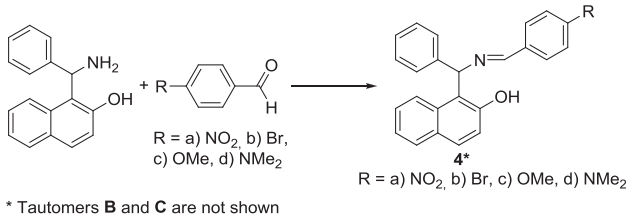


Table 1

Yields, melting points, tautomer ratio in solution of $CDCl_3$ for compounds **3g–3j**.

					Tautomer ratio, %, according to ^{1}H NMR		
Product	R	X	Yield, %	mp °C	**A**	**B**	**C**
3g	H	Br	94	142–143	28	62	10
3h	Me	Br	84	148–150	42	51	7
3i	OMe	Br	91	170–172	58	36	6
3j	NMe_2	Br	86	196–197	92	7	~1

provide full-scale range of variety of these compounds, we obtained a number of model compounds **4a–4d** prepared using standard procedure [4] from unsubstituted 1-(α-aminobenzyl)-2-naphthol and the corresponding *para*-substituted benzaldehydes (Scheme 2). In contrast to compounds **3a–3j**, they do not have a substituent in one of the aryl rings. Compounds **3a–3j** and **4a–4d** were studied in the crystalline phase by vibrational spectroscopy.

It should be noted that the group of German authors [7] ascribe the imine structure to compound **3c** owing to the presence of absorption band at 1618 cm^{-1} in the IR spectra, which they attributed to the valence vibrations of the imino-group. However, according to Smith and Cooper, who published the results of ^{1}H NMR and IR spectroscopic studies of ring-chain tautomerism of some condensation products of racemic 1-α-(aminobenzyl)-2-naphthol with aromatic aldehydes in the solid state [8], all products have a cyclic structure (**B** and **C**). IR spectra of these compounds in KBr pellets had no absorption bands corresponding to the C=N group and contained two sharp bands at 1600–1610 and 1620–1630 cm^{-1}, except the dimethylamino-substituted derivative, which possessed a broad band in the region 1570–1630 cm^{-1}. IR spectra of their solution in chloroform had an additional band at 1650–1660 cm^{-1}, attributed to the C=N group of imine tautomer **A**, which is in equilibrium with *trans*-naphthoxazine (**B**) and *cis*-naphthoxazine (**C**) cyclic tautomers.

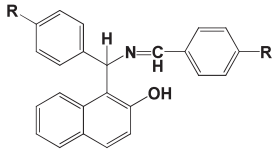
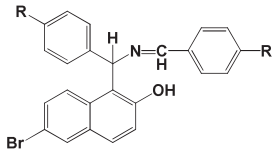
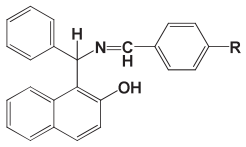
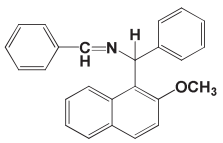
The analysis of the IR spectra of compounds **3a–3j** recorded by us in KBr pellets allows to divide them into two groups (Table 2). The first group (compounds **3a–3d** and **3g–3i**) has two sharp absorption bands at 1587–1598 and 1612–1624 cm^{-1}. The second one (compounds **3e**, **3f**, and **3j**) has three bands in this region: 1575–1579, 1604–1608, and 1637–1646 cm^{-1}. We assign two bands of the first group to the valence C=C vibrations of the naphthol fragment of the oxazine cycle. The third band at 1637–1646 cm^{-1} in the spectra of compounds **3e**, **3f**, and **3j** corresponds to the valence C=N vibrations of the acyclic tautomer and the valence C=C vibration bands being shifted approximately by 5–20 cm^{-1}. Thus, compounds **3a–3d** and **3g–3i** in the solid state have most probably oxazine structure, whereas compounds **3e**, **3f**, and **3j** have imine form.

One more distinctive feature of cyclic tautomers **B** and **C** in comparison with chain form **A** is the sharp band at 3308–3329 cm^{-1} for compounds **3a–3d**, and **3g–3i**, which may be assigned to the valence vibrations of the free NH bond, which is present only in the cyclic oxazine structure and absent in imine form. On the contrary, for compounds **3e**, **3f**, and **3j**, containing electron donor substitutents, the major spectral feature is the broad absorption band at 2000–3000 cm^{-1}, which gives evidence of the strong hydrogen bond of OH group with imine nitrogen O-H···N=C. In the cyclic derivatives, there are no bands in this region of the IR spectra.

Compounds **4a–4d** bearing substituents only in the aromatic fragment at imino-nitrogen have the same spectral features. According to their spectra, *para*-nitro- **4a** and *para*-bromo- **4b** derivatives are cyclic tautomers,

Table 2

Characteristic IR absorption bands (cm^{-1}) of **3a–3j** and **4a–d**, and **5**; 2-naphthol and 2-methoxynaphthalene.

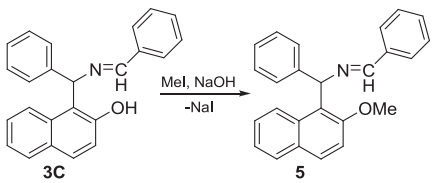
Compound	No.	R	$\nu_{C=N}$	$\nu_{C=C\ arom.}$	$\nu_{NH\text{-}free}$
	**3a**	Br	—	1597, 1624	3311
	3b	Cl	—	1598, 1623	3316
	3c	H	—	1598, 1623	3319
	3d	CH_3	—	1598, 1623	3308
	3e	OCH_3	1646	1575, 1608	—
	3f	$N(CH_3)_2$	1642	1579, 1604	—
	**3g**	H	—	1590, 1618	3311
	3h	CH_3	—	1587, 1613	3311
	3i	OCH_3	—	1588, 1612	3329
	3j	$N(CH_3)_2$	1637	1578, 1605	—
	**4a**	NO_2	—	1598, 1620	3307
	4b	Br	—	1595, 1619	3313
	4c	OCH_3	1641	1580, 1598	—
	4d	$N(CH_3)_2$	1633	1582, 1604	—
	**5**	—	1640	1578, 1596	—
2-naphthol	**1a**	—	—	1600, 1630	—
2-methoxynaphthalene	—	—	—	1597, 1630	—

whereas *para*-methoxy- **4c** and *para*-dimethylamino- **4d** derivatives can be ascribed to acyclic forms (Table 2).

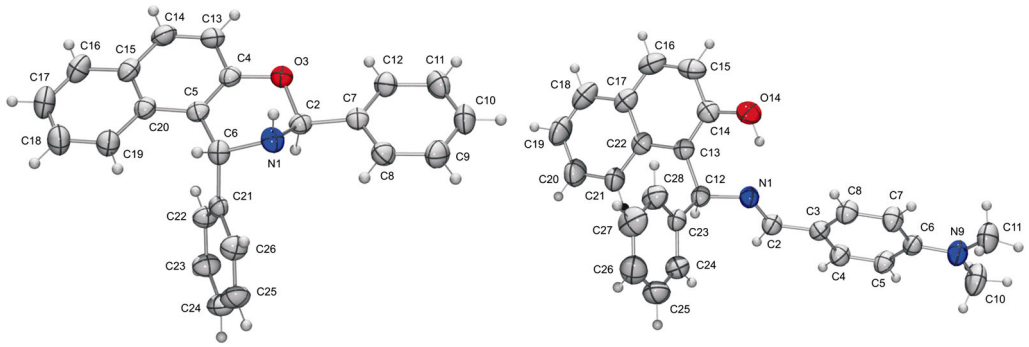
With the purpose to obtain exclusively imine tautomer, we have synthesized O-methylated derivative **5** starting from **3c**.

Compound **5** has been obtained first in multistep synthesis from 1-iodo-2-naphthol [11] and characterized only with melting point and elemental analysis. As an intermediate, it is appeared in the paper [12] without isolation from the reaction mixture and characterization. We have obtained pure **5** in reaction of **3c** with methyl iodide in the presence of alkali (Scheme 3). The IR spectra of compound **5** has an absorption band at 1640 cm^{-1}, characteristic of valence vibrations of the imine group (Table 2), whereas the bands of the aromatic naphthyl fragment are present as the signals at 1578 and 1596 cm^{-1}.

Scheme 3. Preparation of OMe derivative **5**.



Conclusions about the structure of products **3** and **4** in crystalline phase, which we have made on the basis of the IR spectra, were confirmed by X-ray diffraction study of optically active condensation products (***S***)-(+)-**3c** and (***S***)-(+)-**4d**, which were obtained from (*S*)-(+)-1-(α-aminobenzyl)-2-naphthol and benzaldehyde, as well as *p*-dimethylaminobenzaldehyde. Unsubstituted product (***S***)-(+)-**3c**, as well as its bromine derivative, is crystallized as a cyclic isomer (Fig. 1(a)) [13], whereas dimethylaminoderivative (***S***)-(+)-**4d** is acyclic in crystalline phase (Fig. 1(b)). X-Ray study shows that the N-H group in oxazine structure (***S***)-(+)-**3c** is sterically hindered and does



(a) **(b)**

Figure 1. Molecular structure of oxazine (***S***)-(+)-**3c** (a) and imine (***S***)-(+)-**4d** (b).

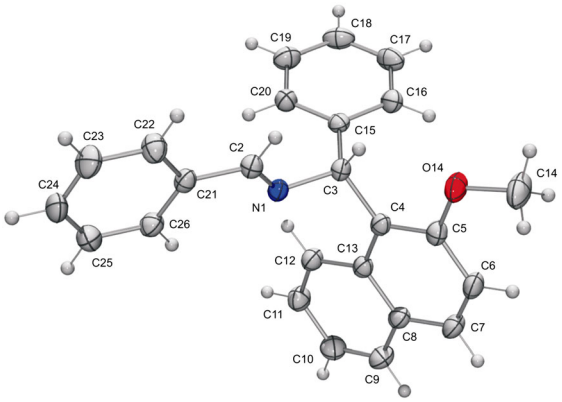


Figure 2. Molecular structure of compound **5**.

not participate in hydrogen bonding. Oxazine structure of the compound (***S***)-(+)-**3c** has a twist conformation with four atoms, O3, C4, C5, and C6 located in the plane of naphthyl fragment, and atoms N1 and C2 moved out on different sides of this plane by approximately equal distance (0.37 Å). Phenyl substituents are in *trans* position to the six-membered oxazine cycle. That is, the *trans* isomer is crystallized, which predominates in solution.

Imine structure (***S***)-(+)-**4d** promotes the formation of an intramolecular hydrogen bond between the naphthyloxy hydrogen atom and imine nitrogen with the formation of the thermodynamically favorable six-membered ring (Fig. 1(b)). The strong intramolecular hydrogen bonds are also evident from the IR spectra in the region 2000–3000 cm^{-1}.

Compound **5** (Fig. 2) has also been studied by X-ray single-crystal diffraction. Because of the absence of the intramolecular hydrogen bond in O-methylated derivative (compared with (***S***)-(+)-**4d** structure, Fig. 1(b)), the naphthyl fragment plane in compound **5** is rotated about C3-C4 bond and is eclipsed with the proton at C3, which minimize the steric interactions of phenyl groups at C2 and C3 with the naphthyl moiety.

CONCLUSIONS

The number of new Betti base derivatives was obtained by the interaction of 6-bromo-2-naphthol and 1,3,5-trisaryl-2,4-diazapenta-1,4-dienes, which shows versatility of proposed method of syntheses of such compounds.

It was established that 1,3-diarylnaphthoxazines might crystallize in acyclic or cyclic forms depending on substituents in aromatic fragments. The increase of the donor ability of substituents in aryl fragments leads to crystallization of these compounds in the form of imine (acyclic) tautomer, which correlates with the enlarged share of imine tautomer in their solutions.

It was shown that analysis of the IR spectra of 1,3-diarylnaphthoxazines bearing substituents in one or in both aryl fragments allows to clearly define their structure in solid state on the basis of characteristic absorption bands corresponding to the C═C, C═N, and N-H groups. These conclusions, made from IR spectra, were unambiguously confirmed by X-ray single-crystal diffraction.

EXPERIMENTAL

^{1}H NMR spectra were recorded on an AVANCE-600 (600.13 MHz) spectrometer (Bruker, Germany) with TMS as the external reference. The following abbreviations are used to indicate multiplicities: s: singlet; m: multiplet. IR spectra were recorded on a Vector 22 spectrometer (Bruker AXS GmbH, Karlsruhe, Germany) from KBr pellets. Melting points were measured on a Boetius melting point microscope.

X-ray data for (***S***)-(+)-**3c**, (***S***)-(+)-**4a** and **5** were collected with a Nonius KappaCCD diffractometer (Bruker AXS GmbH, Karlsruhe, Germany) equipped with a molybdenum fine-focus sealed tube (compound (***S***)-(+)-**3c** was reported earlier by us [14], coordinates of atoms deposited in Cambridge Crystallographic Data Centre: CCDC 628868). Programs used: data collection COLLECT (Nonius B.V., 1998), Denzo-SMN [15], absorption correction SORTAV [16,17], structure solution – SHELXS-97 [18], and structure refinement SHELXL-97 [19]. Crystal data for (***S***)-(+)-**4a**: $C_{26}H_{24}N_2O$, $M = 380.47$, colorless plates, orthorhombic, space group $P2_12_12_1$, $Z = 4$, $a = 6.462(1)$, $b = 16.041(1)$, $c = 19.914(1)$ Å, $V = 2064.2(4)$ $Å^3$, $\rho_{calc} = 1.224\ g \cdot cm^{-3}$, $\mu = 0.75\ cm^{-1}$, $\lambda = 0.71073$ Å, $T = 293(2)$ K, 16432 reflections collected ($\pm h$, $\pm k$, $\pm l$), $[(\sin\theta)/\lambda] = 0.60\ Å^{-1}$,

3758 independent (R_{int} = 0.043) and 2800 observed reflections [$I \geq 2\sigma(I)$], 267 refined parameters, $R1$ = 0.0439, $wR2$ = 0.0828, and goodness of fit 1.165. Crystal data for **5**: $C_{25}H_{21}NO$, M = 351.43, colorless block, monoclinic, space group $P2_1/n$, Z = 4, a = 8.299(1), b = 24.051(1), c = 9.934(1) Å, β = 106.08(3)°, V = 1905.2(3) Å^3, ρ_{calc} = 1.225 g · cm^{-3}, μ = 0.74 cm^{-1}, λ = 0.71073 Å, T = 198(2) K, 27193 reflections collected (±h, ±k, ±l), [(sinθ)/λ] = 0.66 Å^{-1}, 4391 independent (R_{int} = 0.032) and 3233 observed reflections [$I \geq 2\sigma(I)$], 245 refined parameters, $R1$ = 0.0384, $wR2$ = 0.0876, and goodness of fit 1.027. Absolute configuration of (***S***)-(+)-**4a** was not determined but assumed from the initial Betti base. Supplementary crystallographic data sets for the structures of (***S***)-(+)-**4a** and **5** are available through the Cambridge Structural Database with deposition numbers 890339 (for (***S***)-(+)-**4a**) and 890338 (for **5**).

1,3,5-Trisaryl-2,4-diazapenta-1,4-dienes **2a–2f** were prepared according to the known method [20]. 1,3-diarylnaphthoxazines **3a–3j** were obtained by interaction of 2-naphthols **1** with 1,3,5-trisaryl-2,4-diazapenta-1,4-dienes **2** following the method [3]. Tautomeric ratio (**A**, **B**, and **C**) for compounds **3g–3j** was determined by integration of proton signals in ^{1}H NMR spectra and given in Table 1. 1-(α-Aminobenzyl)-naphthol-2 was prepared by the hydrolysis of 1,3-diphenylnaphthoxazine **3c** following the method [21]. Its condensation with a series of substituted benzaldehydes (*p*-nitro-, *p*-bromo-, *p*-methoxy- and *p*-dimethylamino- derivatives) gave oxazines **4a–4d** [4,5]. Compounds (***S***)-(+)-**3c** and (***S***)-(+)-**4a** were obtained following the method [12].

1-[α-(Benzylidene)aminobenzyl]-6-bromonaphth-2-ol (3g). The mixture of 6-bromo-2-naphthol (**1b**, 0.4 g, 1.8 mmoles) and 1,3,5-trisphenyl-2,4-diazapenta-1,4-diene (**2c**, 0.36 g, 1.2 mmoles) in benzene (5 mL) was heated under reflux for 14 h. The precipitated product was filtered off and recrystallized from benzene to give 0.7 g (94%) of **3g**; mp: 142–143°C (ref. [9]; mp: 100°C, ref. [10]; mp: 150–152°C); ^{1}H NMR ($CDCl_3$): δ (tautomer **A**) 6.41 (s, CHN), 8.63 (s, CH═N), (tautomer **B**) 5.63, 5.72 (2s, CHN), (tautomer **C**) 5.86, 5.94 (2s, CHN), (tautomers **A** + **B** + **C**) 7.26–7.96 (m, CH_{arom}); IR (KBr): ν 1590, 1618 (C═C_{arom}), 3320 (NH). *Anal*. Calcd. for $C_{24}H_{18}BrNO$: C, 69.24; H, 4.36; Br, 19.19; N, 3.36. Found: C, 69.03; H, 4.10; Br, 19.45; N, 3.50.

1-[α-(4-Methylbenzylidene)amino-4-methylbenzyl]-6-bromonaphth-2-ol (3h). The mixture of 6-bromo-2-naphthol (**1b**, 0.4 g, 1.8 mmoles) and 1,3,5-tris(4-methylphenyl)-2,4-diazapenta-1,4-diene (**2d**, 0.4 g, 1.2 mmoles) in benzene (5 mL) was heated under reflux for 10 h. The precipitated product was filtered off and recrystallized from benzene to give 0.67 g (84%) of **3h**; mp: 148–150°C; ^{1}H NMR ($CDCl_3$): δ (tautomer **A**) 2.21 (s, Me), 2.33 (s, Me′), 6.25 (s, CHN), 8.47 (s, CH═N), (tautomer **B**) 2.27 (s, Me), 2.29 (s, Me′), 5.49, 5.61 (2s, CHN), (tautomer **C**) 2.23 (s, Me), 2.35 (s, Me′), 5.72, 5.80 (2s, CHN), (tautomers **A** + **B** + **C**) 7.01–7.86 (m, CH_{arom}); IR (KBr): ν 1587, 1613 (C═C_{arom}), 3311 (NH). *Anal*. Calcd. for $C_{26}H_{22}BrNO$: C, 70.28; H, 4.99; Br, 17.98; N, 3.15. Found: C, 69.97; H, 4.70; Br, 17.85; N, 3.08.

1-[α-(4-Methoxybenzylidene)amino-4-methoxybenzyl]-6-bromonaphth-2-ol (3i). The mixture of 6-bromo-2-naphthol (**1b**, 0.4 g, 1.8 mmoles) and1,3,5-tris(4-methoxyphenyl)-2,4-diazapenta-1,4-diene (**2e**, 0.46 g, 1.2 mmoles) in benzene (5 mL) was heated under reflux for 10 h. The precipitated product was filtered off and recrystallized from benzene to give 0.78 g (91%) of **3i**; mp: 170–172°C (ref. [10]; mp: 162–164°C); ^{1}H NMR ($CDCl_3$): δ (tautomer **A**) 3.77 (s, OMe), 3.88 (s, OMe′), 6.32 (s, CHN), 8.53 (s, CH═N), (tautomer **B**) 3.82 (s, OMe), 3.85 (s, OMe′), 5.60, 5.69 (2s, CHN), (tautomer **C**) 3.79 (s, OMe), 3.93 (s, OMe′), 5.81, 5.89 (2s, CHN), (tautomers **A** + **B** + **C**) 6.83–7.96 (m, CH_{arom}); IR (KBr): ν 1588, 1612 (C═C_{arom}), 3329 (NH). *Anal*. Calcd. for $C_{26}H_{22}BrNO_3$: C, 65.55; H, 4.66; Br, 16.77; N, 2.94. Found: C, 65.61; H, 4.71; Br, 16.90; N, 3.01.

1-[α-(4-Dimethylaminobenzylidene)amino-4-dimethylaminobenzyl]-6-bromonaphth-2-ol (3j). The mixture of 6-bromo-2-naphthol (**1b**, 0.4 g, 1.8 mmoles) and 1,3,5-tris (4-dimethylaminophenyl)-2,4-diazapenta-1,4-diene (**2f**, 0.51 g, 1.2 mmoles) in benzene (5 mL) was heated under reflux for 6 h. The precipitated product was filtered off and recrystallized from benzene to give 0.77 g (86%) of **3j**; mp: 196–197°C (ref. [9]; mp: 120°C); ^{1}H NMR ($CDCl_3$): δ (tautomer **A**) 2.89 (s, NMe_2), 3.04 (s, NMe_2'), 6.24 (s, CHN), 8.41 (s, CH═N), (tautomer **B**) 2.95 (s, NMe_2), 2.97 (s, NMe_2'), 5.57, 5.71 (2s, CHN), (tautomer **C**) 5.78, 5.85 (2s, CHN), (tautomers **A** + **B** + **C**) 6.63–7.91 (m, CH_{arom}); IR (KBr): ν 1578, 1605 (C═C_{arom}), 1637 (C═N). *Anal*. Calcd. for $C_{28}H_{28}BrN_3O$: C, 66.93; H, 5.62; Br, 15.90; N, 8.36. Found: C, 66.71; H, 5.53; Br, 16.08; N, 8.48.

1-(α-Benzylidenaminobenzyl)-2-methoxynaphthaline (5). The compound was obtained by the method described in [12] with our modifications. 1,3-diphenylnaphthoxazine (**3c**, 10.12 g, 0.03 mole) was dissolved in mixture of dry dioxane (60 mL) and dry acetone (80 mL) under heating till 50°C. The solution was cooled to 0°C, and powdered NaOH (1.8 g, 0.045 mole) was added. Afterwards, methyl iodide (11.45 g, 0.08 mole) was added dropwise at 0°C. After the addition of methyl iodide, the reaction mixture was stirred at room temperature for 8 h. The next day, the precipitate of NaI was filtered off and washed with ethyl acetate (50 mL × 2), and the filtrate was dried over Na_2SO_4 and combined with mother liquor. Solvents were removed in vacuo, and resulting dense residue was extracted by absolute ethyl alcohol (80 mL) at 60°C. The obtained precipitate was separated and extracted with diethyl ether. The ether extract was separated, and on cooling, it dropped out of the crystalline product **5**. The latter was filtered off. Extraction was repeated until complete isolation of the product to give 6.2 g (59%) of **5**. Analytically pure sample was obtained by recrystallization from ether. mp: 106–108°C (ref. [11]; mp: 104–106°C); ^{1}H NMR ($CDCl_3$): δ 4.03 (s, 3H, OCH_3), 6.91 (s, 1H, CHN), 7.19–8.24 (m, 16H, CH_{arom}), 8.49 (s, 1H, CH═N); IR (cm^{-1}): ν 1578, 1596 (C═C_{arom}), 1640 (C═N). *Anal*. Calcd. for $C_{25}H_{21}NO$: C, 85.44; H, 6.02; N, 3.99. Found: C, 85.05; H, 6.08; N, 4.12.

Acknowledgments. This work was supported by the Russian Foundation for Basic Research (grants no. 11-03-00463 and 11-03-90449) and by the Ministry of Education and Science of the Russian Federation (grant no. 8446).

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