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A simple route to 2-aryl-substituted naphtho[2,1-*e*][1,2,4]-triazinium and naphtho[2,1-*e*][1,2,3,4]tetrazinium salts from 1-arylazo-substituted 2-naphthylamines

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Abstract: 1-Arylazo-substituted 2-naphthylamines, which are easily obtainable by the coupling of arene diazonium salts with 2-aminonaphthalene-sulfonic acid, can be transformed by reaction with reactive carboxylic acid derivatives or nitrosation reagents into novel 2-aryl-substituted naphtho[2,1-*e*][1,2,4]triazinium and naphtho[2,1-*e*][1,2,3,4]tetrazinium salts, respectively.

Keywords: 2-naphthylamine-1-azo compounds; acylation; naphtho[2,1-*e*][1,2,3,4]tetrazinium salts; naphtho[2,1-*e*]-[1,2,4]triazinium salts; nitrosation.

1 Introduction

2-Amino azoarenes **3**, which are obtainable by the coupling of a 4-substituted aniline **1** with an arene diazonium salt **2** [1], are interesting starting materials for the synthesis of certain heterocyclic systems. Thus, these compounds **3** can be transformed by oxidation into benzo[1,2,3]triazines **4** [2] or by reaction with nitrosation reagents NOX into rather unstable diazonium salts **5**, which give rise by their reaction with special nucleophiles to 1-imino-benzo[1,2,3] triazolium ylides **6** [3]. As coupling of the anilines **1** with the arene diazonium salts **2** gives rise usually to low yields of products [4, 5], the heterocyclic compounds **4** and **6** prepared this way from **3** have been documented only in a few examples (Scheme 1).

Since the 1-arylazo-2-naphthylamines **8** are, as benzo-condensed 2-amino-substituted azoarenes, much more easily available, e.g. by the coupling of 2-naphtylamine-1-sulfonic acid **7** with arenediazonium salts **2** [6], their use as starting materials for the synthesis of heterocyclic compounds was studied by us in more detail. Some of the results obtained are reported in the following and depicted in Scheme 2.

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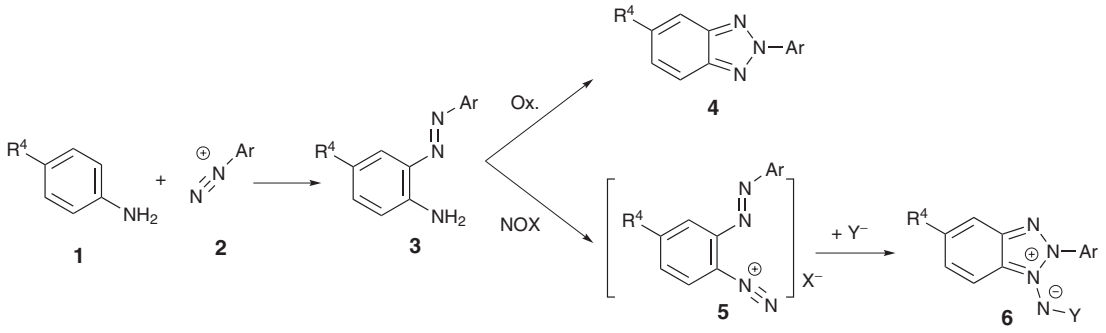
2 Results and discussion

Besides the transformation of 1-arylazo-2-naphthylamines **8** into the corresponding naphtho[1,2,3]triazoles, which was studied earlier by other authors [7], the reaction of azo compounds **8** with certain reactive carboxylic acid derivatives, such as the Vilsmeier reagent or carboxylic acid anhydrides, has been studied. Thereby, novel naphtho[1,2,4]triazinium salts **9** and **10** are formed in mostly satisfactory yields.

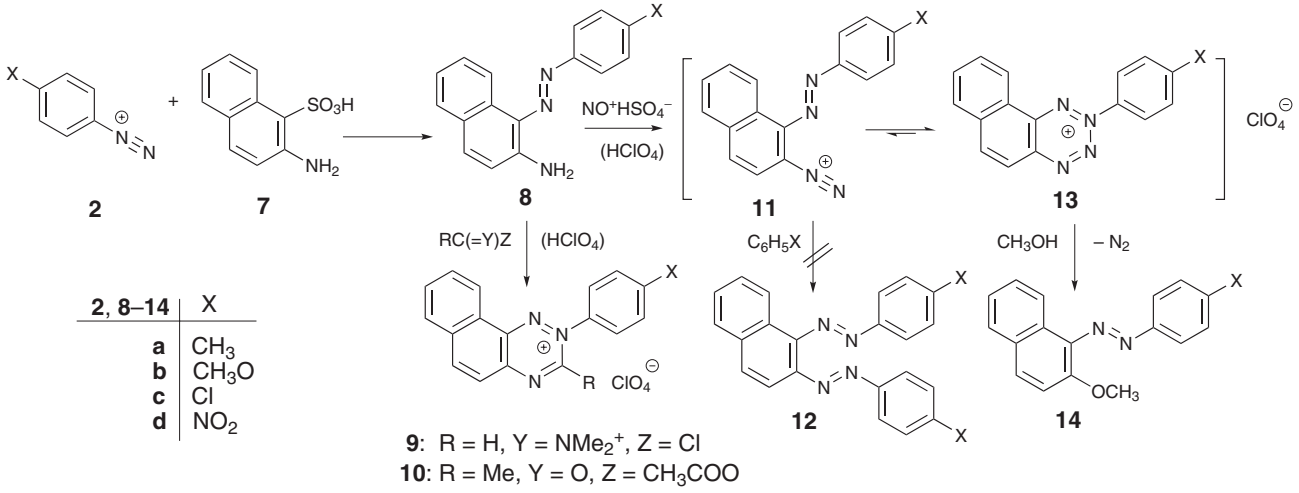
Furthermore, the 1-arylazo-2-naphthylamines **8** are able to react with certain nitrosation reagents, such as nitrosyl sulfuric acid, yielding the corresponding diazonium salts **11**. These salts exist, however, as confirmed by IR and ^{1}H NMR measurements, in a valence tautomeric naphtha[1,2,3,4]tetrazinium structure **13** and are therefore not able to couple with reactive aromatic compounds C_6H_5X, such as *N*,*N*-dialkylanilines (X=NR_2) or phenols (X=OH), to give the corresponding bis-azo compounds **12**. Instead of coupling, the naphtho[1,2,3,4]tetrazinium salts **13** decompose by standing, and yield, e.g. in methanolic solution, the corresponding 2-methoxy-1-arylazonaphthalenes **14**.

The structures of the compounds prepared were confirmed by mass spectrometry and ^{1}H NMR spectroscopy. Thus, in the mass spectra of compounds **10**, the expected molecular ions were detected. In the mass spectra of the salts **13**, besides the molecular ion peaks, further peaks were observed, indicating a loss of N_2. In the mass spectra of some of the salts **9**, adduct peaks with methanol could be detected next to those of the corresponding molecular ions. Obviously, the methanol peaks stemmed from the solvent used for measuring the mass spectra and was added to the corresponding salt peaks during the measurement. In the ^{1}H NMR spectra of the salts **9** and **10**, characteristic signals for the protons at the 1,2,3-triazine rings at about 10.0 ppm and for the methyl group at about 3.0 ppm, respectively, were found.

To exclude diazonium salts **11** as the products obtained by the diazotation of compounds **8**, IR spectra



Scheme 1: Reported use of 2-aminoazobenzenes **3** for the preparation of heterocyclic compounds.



Scheme 2: Synthetic route to 1-arylazo-2-naphthylamines **8** and their transformation into new 2-aryl-substituted naphtho[2,1-*e*][1,2,4] triazinium salts **9** and **10**, and naphtha[1,2-*e*][1,2,3,4]tetrazinium salts **13**.

of these compounds were recorded. In no case, bands in the region 2250–2300 cm^{-1}, which are characteristic for the $-\overset{\oplus}{N}\equiv N$ moiety, were found. Therefore, the products obtained by this reaction exist exclusively in their heterocyclic 1,2,3,4-tetrazinium form **13**.

3 Experimental section

^{1}H NMR spectra were recorded with a Bruker DRX 500 P instrument at 500.13 MHz; chemical shifts δ are given in ppm and coupling constants (J) in Hz. UV/vis spectra were measured in dichloromethane with a Perkin Elmer Lambda 900 UV/vis/NIR spectrometer, and mass spectra were recorded with a Bruker Esquire MS (ESI) or with a MAT 8200 Finnigan spectrometer (HRMS). Melting points were measured with a Boetius heating-table microscope.

3.1 Preparation of 1-arylazo-substituted 2-napthylamines 8 (general procedure)

To an aromatic or heteroaromatic amine (10 mmol) dissolved in aqueous hydrochloric acid (15 mL, 50%), sodium nitrite (0.84 g, 8 mmol) was added in small portions at 0°C. After 30 min, urea (1 g) was added, and the resulting mixture was poured into an aqueous/methanolic solution (1:1, 50 mL) of 2-aminonaphthalene sulfonic acid (2.2 g, 10 mmol). The product precipitated was isolated by suction, washed with water, and recrystallized from butanol after drying.

The following 1-arylazo substituted 2-naphthylamines **8** were prepared by the above procedure:

3.1.1 1-(4-Tolylazo)-2-naphthylamin (8a)

This compound was prepared from 2-amino-naphthaline-1-sulfonic acid (**7**) and 4-toluene diazonium hydrosulfate

(**2a**) as an orange crystalline powder; yield 80%, m.p. 111°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 436 nm (4.34). ^{1}H NMR ([D_6]DMSO): δ = 3.84 (s, 3H, CH_3), 7.10 (m, 2 arom. H), 7.29 (d, J = 8.0 Hz, 1 arom. H), 7.50 (d, J = 8.0 Hz, 1 arom. H), 7.82 (d, J = 9.0 Hz, 2 arom. H), 7.92 (d, J = 8.0 Hz, 2 arom. H), 8.72 (d, J = 7.5 Hz, 2 arom. H), 8.05 (d, J = 9.5 Hz, 1 arom. H) ppm. MS ((+)-ESI): m/z = 262.0 (calcd. 262.13 for $[C_{17}H_{16}N_3]^+$).

3.1.2 1-(4-Methoxyphenylazo)-2-naphthylamin (8b)

It was prepared from 2-amino-naphthaline-1-sulfonic acid (**7**) and 4-methoxybenzene diazonium hydrosulfate (**2b**) as an orange crystalline powder; yield 93%, m.p. 126°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 451 nm (4.37). ^{1}H NMR ([D_6] DMSO): δ = 2.26 (s, 3H, CH_3), 6.84 (d, J = 10.0 Hz, 1 arom. H), 7.19 (d, J = 9.0 Hz, 2 arom. H), 7.36 (d, J = 9.0 Hz, 2 arom. H), 7.44 (t, J = 7.0 Hz, 1 arom. H), 7.60 (t, J = 8.0 Hz, 1 arom. H), 7.72 (d, J = 10.0 Hz, 1 arom. H), 8.05 (d, J = 8.0 Hz, 1 arom. H) ppm. MS ((+)-ESI): m/z = 278.1 (calcd. 278.13 for $[C_{17}H_{16}N_3O]^+$).

3.1.3 1-(4-Chlorphenylazo)-2-naphthylamin (8c)

It was prepared from 2-amino-naphthaline-1-sulfonic acid (**7**) and 4-chlorobenzene diazonium hydrosulfate (**2c**) as a brown crystalline powder; yield 77%, m.p. 112°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 457 nm (4.28). ^{1}H NMR ([D_6]DMSO): δ = 7.15 (d, J = 9.0 Hz, 1 arom. H), 7.33 (m, 1 arom. H), 7.51–7.56 (m, 1 arom. H), 7.62 (d, J = 8.0 Hz, 2 arom. H), 7.73–7.80 (d, J = 9.0 Hz, 2 arom. H), 7.98 (d, J = 9.0 Hz, 2 arom. H), 8.72 (d, J = 9.0 Hz, 1 arom. H) ppm. MS ((+)-ESI): m/z = 282.1 (calcd. 282.74 for $[C_{16}H_{13}ClN_3]^+$).

3.1.4 1-(4-Nitrophenyl-2-ylazo)-2-naphthylamin (8d)

It was prepared from 2-amino-naphthaline-1-sulfonic acid (**7**) and 4-nitrobenzene diazonium hydrosulfate (**2d**) as a red crystalline powder; yield 95%, m.p. 185–187°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 503 nm (4.47). ^{1}H NMR ([D_6] DMSO): δ = 7.16 (d, J = 9.0 Hz, 1 arom. H), 7.35 (m, 1 arom. H), 7.56 (m, 1 arom. H), 7.74 (d, J = 8.0 Hz, 1 arom. H), 7.82 (s, J = 9.0 Hz, 1 arom. H), 8.12 (d, J = 9.0 Hz, 2 arom. H), 8.36 (d, J = 9.0 Hz, 2 arom. H), 8.71 (d, J = 8.0 Hz, 1 arom. H) ppm. MS ((+)-ESI): m/z = 293.1 (calcd. 293.10 for $[C_{13}H_{11}N_4S]^+$).

3.2 Preparation of 2-aryl-substituted naphtho[2,1-*e*][1,2,4]triazinium perchlorates (9) (general procedure)

To a solution of a 1-arylazo substituted 2-napthylamine **8** (10 mmol) in DMF (20 mL), phosphoryl chloride (5 mL) was added with cooling at ~0°C. After allowing it to stand for some time, methanol (15 mL) was added, followed by perchloric acid (5 mL, 70%) and diethyl ether (50 mL). The precipitate formed was isolated by suction, dried under air, and recrystallized from acetic acid/ethyl acetate.

The following 3-aryl-substituted naphtho[1,2,4]-triazinium perchlorates (**9**) were prepared by the above procedure.

3.2.1 2-(*p*-Tolyl)naphtho[2,1-*e*][1,2,4]triazinium perchlorate (9a)

It was prepared from 1-(4-tolylazo)-2-naphthylamine (**8a**) as an orange crystalline powder; yield 65%, m.p. 252–254°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 459 nm (4.18). ^{1}H NMR (TFA): δ = 2.49 (s, 3H, CH_3), 7.56 (d, J = 8.0 Hz, 2 arom. H), 7.93 (d, J = 8.0 Hz, 2 arom. H), 8.08–8.13 (m, 3 arom. H), 8.21 (d, J = 8.0 Hz, 1 arom. H), 8.84 (d, J = 8.0 Hz, 1 arom. H), 9.28 (t, J = 8.0 Hz, 1 arom. H), 10.33 (s, 1 arom. H) ppm. MS ((+)-ESI): m/z = 304.1 (calcd. 304.37 for $[C_{18}H_{14}N_3 + CH_3OH]^+$).

3.2.2 2-(4-Methoxyphenyl)naphtho[2,1-*e*][1,2,4]-triazinium perchlorate (9b)

It was prepared from 1-(4-methoxyphenylazo)-2-naphthylamine (**8b**) as an orange crystalline powder; yield 66%, m.p. 178–180°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 484 nm (4.16). ^{1}H NMR (TFA): δ = 3.95 (s, 3H, OCH_3), 7.26 (d, J = 15.0 Hz, 2 arom. H), 8.04–8.18 (m, 6 arom. H), 8.78 (d, J = 15 Hz, 1 arom. H), 9.25–9.27 (m, 1 arom. H), 10.31 (s, 1 hetarom. H) ppm. MS ((+)-ESI): m/z = 288.1 (calcd. 288.11 for $[C_{18}H_{14}N_3O]^+$).

3.2.3 2-(4-Chloropheny)naphtho[2,1-*e*][1,2,4]triazinium perchlorate (9c)

It was prepared from 1-(4-chlorophenylazo)-2-naphthylamine (**8c**) as a brown crystalline powder; yield 74%, m.p. 158–160°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 456 nm (4.00). ^{1}H NMR (TFA): δ = 7.68 (d, J = 10.0 Hz, 2 arom.

H), 7.98 (d, J = 10 Hz, 2 arom. H), 8.05–8.10 (m, 3 arom. H), 8.16 (d, J = 10.0 Hz, 1 arom. H), 8.81 (d, J = 10.0 Hz, 1 arom. H), 9.23 (d, J = 5.0 Hz, 1 arom. H), 10.31 (s, hetarom. H) ppm. MS ((+)-ESI): m/z = 324.2 (calcd. 324.09 for $[C_{17}H_{11}ClN_3 + CH_3OH]^+$).

3.2.4 2-(4-Nitrophenyl)naphtho[2,1-*e*][1,2,4]triazinium perchlorate (9d)

It was prepared from 1-(4-nitrophenylazo)-2-naphthylamine (**8d**) as a brown crystalline powder; yield 32%, m.p. = 176–180°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 452 nm (4.00). ^{1}H NMR (TFA): δ = 8.14–8.23 (m, 3 arom. H), 8.26 (d, J = 6.0 Hz, 1 arom. H), 8.37 (d, J = 13 Hz, 2 arom. H), 8.63 (d, J = 14 Hz, 2 arom. H), 8.93 (d, J = 15 Hz, 1 arom. H), 9.28 (d, J = 8 Hz, 1 arom. H), 10.51 (s, 1 hetarom. H) ppm. MS ((+)-ESI): m/z = 335.1 (calcd. 335.11 for $[C_{14}H_9N_4S + CH_3OH]^+$).

3.3 Preparation of 2-aryl-substituted 3-methylnaphtho[2,1-*e*][1,2,4]triazinium salts (10) (general procedure)

To a solution of a 1-arylazo-substituted 2-napthylamine **8** (10 mmol) in acetic anhydride (15 mL), magnesium perchlorate (3 g) was added with cooling. After allowing it to stand for some time, the mixture was heated at 100°C until a clear solution was obtained. Following cooling down to room temperature, ethyl acetate (50 mL) was added, and the product precipitated was isolated by suction, dried, and recrystallized from acetic acid.

The following 3-(het)aryl-substituted 3-methylnaphtho[2,1-*e*][1,2,4]triazinium perchlorates (**10**) were prepared by this procedure:

3.3.1 3-Methyl-2-(*p*-tolyl)naphtho[2,1-*e*][1,2,4]triazinium perchlorate (10a)

It was prepared from 1-(4-tolylazo)-2-naphthylamine (**8a**) as a yellow crystalline powder; yield 71%, m.p. 245°C (dec.). UV/vis (CH_2Cl_2): λ_{max} (log ε): = 431 nm (3.94). ^{1}H NMR (TFA): δ = 2.46 (s, 3H, CH_3), 3.06 (S, 3H, CH_3), 7.48–7.52 (m, 4 arom. H), 7.93–7.96 (m, 1 arom. H), 8.0 (d. J = 8.0 Hz, 2 arom. H), 8.72 (d, J = 8.0 Hz, 1 arom. H9), 9.04 (d, J = 8.0 Hz, 1 arom. H) ppm. MS ((+)-ESI): m/z = 286.1 (calcd. 286.13 for $[C_{19}H_{16}N_3]^+$).

3.3.2 2-(4-Methoxyphenyl)-3-methylnaphtho[2,1-*e*]-[1,2,4]triazinium perchlorate (10b)

It was prepared from 1-(4-methoxyphenylazo)-2-naphthylamine (**8b**) as a yellow crystalline powder; yield 86%, m.p. 229°C (dec.). UV/vis (CH_2Cl_2): λ_{max} (log ε): = 439 nm (4.00). ^{1}H NMR ($CDCl_3$): δ = 3.22 (s, 3H, CH_3), 4.09 (s, 3H, CH_3O), 7.38 (d, J = 9.0 Hz, 2 arom. H), 7.76 (d, J = 9.0 Hz, 2 arom. H), 8.08–8.16 (m, 3 arom. H), 8.23 (d, J = 8.0 Hz, 1 arom. H), 8.86 (d, J = 9.0 Hz, 1 arom. H), 9.18 (d, J = 8.0 Hz, 1 arom. H) ppm. MS ((+)-ESI): m/z = 302.1 (calcd. 302.13 for $[C_{19}H_{16}N_3O]^+$).

3.3.3 2-(4-Chlorophenyl)-3-methylnaphtho[2,1-*e*][1,2,4]-triazinium perchlorate (10c)

It was prepared from 1-(4-chlorophenylazo)-2-naphthylamine (**8c**) as a yellow crystalline powder; yield 88%, m.p. 214°C (dec.). UV/vis (CH_2Cl_2): λ_{max} (log ε): = 432 nm (3.86). ^{1}H NMR ($CDCl_3$): δ = 3.06 (s, 3H, CH_3), 7.61 (d, J = 9.0 Hz, 2 arom. H), 7.66 (d, J = 8.0 Hz, 2 arom. H), 7.94–8.03 (m, 3 arom. H), 8.10 (d, J = 8.0 Hz, 1 arom. H), 8.74 (d, J = 9.0 Hz, 1 arom. H), 9.02 (d J = 8.0 Hz, 1 arom. H) ppm. MS ((+)-ESI): m/z = 338.2, (calcd. 338.11 for $[C_{18}H_{13}ClN_3 + CH_3OH]^+$).

3.3.4 3-Methyl-2-(4-nitrophenyl)naphtho[2,1-*e*][1,2,4]-triazinium perchlorate (10d)

It was prepared from 1-(4-nitrophenylazo)-2-naphthylamine (**8d**) as a brown crystalline powder; yield 50%, m.p. 227–229°C (dec.). UV/vis (CH_2Cl_2): λ_{max} (log ε): = 438 nm (3.82). ^{1}H NMR ($CDCl_3$): δ = 3.08 (s, 3H, CH_3), 7.96 (t, 1 arom. H), 8.00–8.04 (m, 4 arom. H), 8.12 (d, J = 8.0 Hz, 1 arom. H), 8.59 (d, J = 8.5 Hz, 2 arom. H), 8.78 (d, J = 9.0 Hz, 1 arom. H), 9.00 (d, J = 8.0 Hz, 1 arom. H) ppm. MS ((+)-ESI): m/z = 349.2 (calcd. 349,13 for $[C_{15}H_{11}N_4S + CH_3OH]^+$).

3.4 Preparation of 2-aryl-substituted naphtho[1,2-*e*][1,2,3,4]-tetrazinium salts (13) (general procedure)

To a solution of a 1-arylazo-substituted 2-napthylamine **8** (10 mmol) in ethyl acetate (50 mL) containing magnesium perchlorate (3 g), isoamyl nitrite (2.3 g, 20 mmol) was added at 0°C. Subsequently, after some time, perchloric acid (5 mL, 70%) was added to the reaction mixture at 0°C. After warming the mixture to room temperature, the

product precipitated, which was isolated by suction and recrystallized after drying.

The following 2-(het)aryl-substituted naphtho[1,2-*e*][1,2,3,4]tetrazinium perchlorates were prepared by the above procedure:

3.4.1 2-(*p*-Tolyl)naphtho[1,2-*e*][1,2,3,4]tetrazinium perchlorate (13a)

It was prepared from 1-(*p*-tolylazo)-2-naphtylamine (**8a**) as a red solid; yield 56%, m.p. 189–190°C (dec.). UV/vis (CH_2Cl_2): λ_{max} (log ε): = 502 nm (4.35). ^{1}H NMR ($CDCl_3$): δ = 2.50 (s, 3H, CH_3), 7.55 (d, J = 8.5 Hz, 2 arom. H), 8.16–8.26 (m, 2 arom. H), 8.43 (d, J = 9.0 Hz, 1 arom. H), 8.49 (d, J = 8.5 Hz, 2 arom. H), 8.91 (d, J = 7.0 Hz, 1 arom. H), 9.41 (d, J = 8.0 Hz, 1 arom. H) ppm. HRMS ((+)-ESI): m/z = 273.1138 (calcd. 273.1140 for $[C_{17}H_{13}N_4H]^+$).

3.4.2 2-(4-Methoxyphenyl)naphtho[1,2-*e*][1,2,3,4]-tetrazinium perchlorate (13b)

It was prepared from 1-(4-methoxyphenylazo)-2-naphtylamine (**8b**) as a red solid; yield 64%, m.p. 178–180°C (dec.). UV/vis (CH_2Cl_2): λ_{max} (log ε): = 535 nm (4.22).^{1}H NMR (TFA): δ = 4.00 (s, 3H, OCH_3), 7.24 (d, J = 15 Hz, 2 arom. H), 8.11–8.27 (m, 3 arom. H), 8.32 (d, J = 15 Hz, 1 arom. H), 8.56 (d, J = 15 Hz, 2 arom. H), 8.74 (d, J = 15 Hz, 1 arom. H), 9.33 (d, J = 14 Hz, 1 arom. H) ppm. HRMS ((+)-ESI): m/z = 289.1086 (calcd. 289.1089 for $[C_{17}H_{13}N_4O]^+$).

3.4.3 2-(4-Chlorophenyl)naphtho[1,2-*e*][1,2,3,4]-tetrazinium perchlorate (13c)

It was prepared from 1-(*p*-chlorophenylazo)-2-naphtylamine (**8c**) as a brown solid; yield 84%, m.p. 185°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 494 nm (4.08). ^{1}H NMR (TFA): δ = 7.70 (d, J = 9.0 Hz, 2 arom. H), 8.17 (t, J = 7.0 Hz, 1 arom. H), 8.22 (t, J = 7.0 Hz, 1 arom. H), 8.29 (d, J = 8.0 Hz, 1 arom. H), 8.42 (d, J = 9.0 Hz, 1 arom. H), 8.46 (d, J = 9.0 Hz, 2 arom. H), 8.83 (d, J = 9.0 Hz, 1 arom. H), 9.26 (d, J = 8.5 Hz, 1 arom. H) ppm. HRMS ((+)-ESI): m/z = 293.0594 (calcd. 293.0594 for $[C_{16}H_{10}ClN_4]^+$).

3.4.4 2-(4-Nitrophenyl)naphtho[1,2-*e*][1,2,3,4]-tetrazinium perchlorate (13d)

It was prepared from 1-(4-nitrophenylazo)-2-naphthylamine (**8d**) as a red solid; yield 54%, m.p. 173°C. UV/vis (CH_2Cl_2): λ_{max} (log ε): = 494 nm (3.91). ^{1}H NMR (TFA): δ = 8.09–8.19 (m, 2 arom. H), 8.25–8.33 (m, 1 arom. H), 8.41 (d, J = 15 Hz, 1 arom. H), 8.55 (s, 4 arom. H), 8.68 (d, J = 14 Hz, 1 arom. H), 9.28 (d, J = 8 Hz, 1 arom. H) ppm. HRMS ((+)-ESI): m/z = 304.0833 (calcd. 304.0834 for $[C_{16}H_{10}N_5O_2]^+$).

3.4.5 2-Methoxy-1-(4-tolylazo)naphthalene 14a (R=Me)

It was prepared by letting a methanolic solution of 2-(tolyl)naphtho[1,2-*e*][1,2,3,4]tetrazinium perchlorate (**13a**) stand for 3 days at room temperature and by adding water. The product precipitated was isolated by filtration in a yield of 68%. After drying under air and recrystallization, an orange solid with a melting point of 63°C (Lit. [8]: m.p. 61–63°C) was obtained. ^{1}H NMR ($CDCl_3$): δ = 2.50 (s, 3H, CH_3), 4.46 (s, 3H, OCH_3), 7.41 (d, J = 5.0 Hz, 2 arom. H), 7.46 (d, J = 8.0 Hz, 1 arom. H), 7.60 (t, J = 7.0 Hz, 1 arom. H), 7.84 (t, J = 7.0 Hz, 1 arom. H), 7.84–7.86 (m, 3 arom. H), 8.40 (d, J = 10.0 Hz, 1 arom. H), 8.70 (d, J = 10.0 Hz, 1 arom. H). MS ((+)-ESI): m/z = 277.2 (calcd. 277.13 for $[C_{18}H_{17}N_2O]^+$).

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