peri-Naphthylenediamines
22.* Synthesis of 1,4,5-tris(dimethylamino)naphthalene and other "proton sponge" 4-amino derivatives

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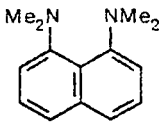
Catalytic hydrogenation of 4-nitro-1,8-bis(dimethylamino)naphthalene afforded the previously unknown 1-amino-4,5-bis(dimethylamino)naphthalene, subsequent methylation of which gave 1-methylamino and 1-dimethylamino "proton sponge" derivatives. The pK_a values of 1-amino-, 1-methylamino-, and 1-dimethylamino-4,5-bis(dimethylamino)-naphthalenes estimated by ^1H NMR in DMSO-d_6 are equal to 9.8, 10.1, and 8.0, respectively.

Key words: naphthylamines, "proton sponge", catalytic hydrogenation, methylation, basicity, conformational isomerism.

It is known that the characteristic feature of 1,8-bis(dimethylamino)naphthalene (**1**) ("proton sponge") is the fact that its basicity is unusually high for amines: its pK_a is 12.34 in water,[2] 18.18 in acetonitrile,[3] and 7.5 in DMSO.[4] The basicity of the recently synthesized 1,4,5,8-tetrakis(dimethylamino)naphthalene (**2**) (the "double proton sponge") measured[5] by the competition method in DMSO is 2.3 orders of magnitude higher than that of compound **1**: pK_a = 9.8. The purpose of this work was to synthesize an intermediate member of this series, 1,4,5-tris(dimethylamino)naphthalene (**3**), which was logically named a "sesquisponge". Its precursor, the previously unknown 1-amino-4,5-bis(dimethylamino)-naphthalene (**4**), was obtained by us in quantitative yield



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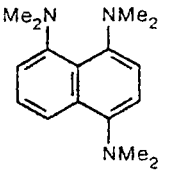

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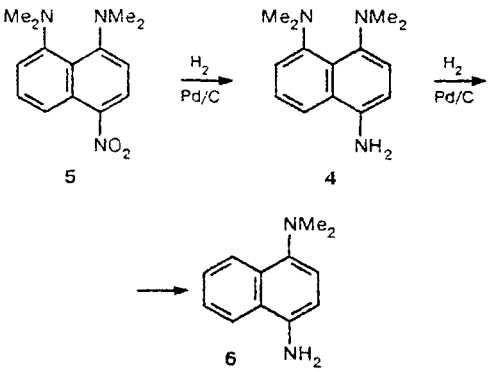
Scheme 1



by catalytic hydrogenation of the readily available 4-nitro-1,8-bis(dimethylamino)naphthalene (**5**)[6] (Scheme 1). We were unable to reduce the latter compound to amine with sodium dithionate in an alcoholic ammonium medium or with $NaBH_4$ in alcohol or in acetic acid.

Amine **4** is an oily compound that is readily oxidized in the air. A peculiar feature of its ^1H NMR spectrum (Table 1) is the long-range spin-spin coupling between the H-2 and NH_2 protons with a surprisingly large constant, $^4J_{NH_2,H\text{-}2}$ = 2.6 Hz. The H-2 signal is a complex doublet.

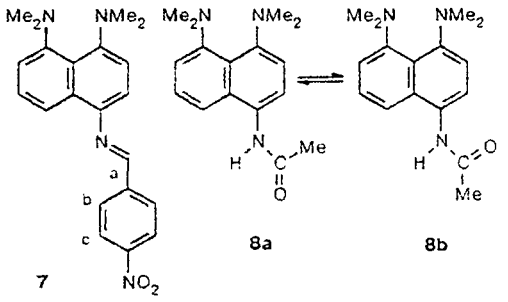
Increasing the hydrogenation time to 30 h or prolonged (up to 7 days) storage of the mixture over a catalyst resulted in the elimination of the 8-NMe_2 group

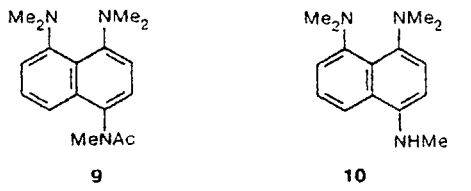
*For Part 21, see Ref. 1.
Part 19 (*Izv. Akad. Nauk, Ser. Khim.*, 1997, 348 [*Russ. Chem. Bull.*, 1997, **46**, 334 (Engl. Transl.)]) contains missprints. The correct variant is as follows.
Di[4,5-bis(dimethylamino)naphthyl-1]methane (5). ^1H NMR ($CDCl_3$), δ: 2.77 (s, 6 H, 4-Me_2N); 2.83 (s, 6 H, 5-Me_2N); 4.62 (s, 2 H, CH_2); 6.80 (d, 1 H, H(3), $J_{3,2}$ = 7.8 Hz); 6.90 (d, 1 H, H(2)); 6.96 (dd, 1 H, H(6), $J_{6,7}$ = 7.51, $J_{6,8}$ < 1Hz); 7.31 (t, 1 H, H(7), $J_{7,5}$ = 7.62, $J_{7,8}$ = 8.17Hz); 7.57 (dd, 1 H, H(8), $J_{8,7}$ = 8.10, $J_{8,6}$ < 1Hz).

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya*, No. 8, pp. 1501—1504, August, 1997.

affording *N,N*-dimethyl-1,4-naphthylenediamine (**6**) in a quantitative yield.

The amino derivative of the "proton sponge" (**4**) exhibits properties of a typical arylamine. For example, its reaction with *p*-nitrobenzaldehyde gives brightly colored azomethine **7** in 81% yield, while its treatment with acetic anhydride results in *N*-acetyl derivative **8**. In chloroform at 20 °C, the latter exists as a mixture of two rotamers **8a** and **8b** in a 2 : 3 ratio, which can be explained by the existence of a significant barrier to rotation around the N—C(O) bond.





It is known[7] that *N*-substituted acetamides exist primarily in the *Z*-conformation of the type **8b**. This is likely to be true for compound **8** too, in whose ^{1}H NMR spectrum in $CDCl_3$ the signal of the NH group proton of rotamer **8a** (δ 7.24) is located in a weaker field than the signal of the NH group proton of rotamer **8b** (δ 7.07); the opposite is observed for the H-2 signals (δ 7.18 and 7.35, respectively).

The splitting of signals in the ^{1}H NMR spectrum caused by conformational transitions is absent for the acetamido derivative **8** in DMSO-d_6. It is known[8] that rotation around an amide bond becomes impeded as the polarity of the solvent increases and, to a greater extent, as its ability to form H-bonds increases. As a result, stabilization of the more thermodynamically stable rotamer, probably **8b**, takes place in DMSO.

An attempt at diazotization of compound **4** was not quite successful. In particular, treatment of **4** with the HBr—$NaNO_2$ system followed by heating in the presence of Cu_2Br_2 resulted in strong resinification, along with the formation of the corresponding bromo derivative (no more than 25%). Such a low yield of the bromide can be explained by the high reactivity of amine **4** and its tendency to undergo oxidation. The occurrence of competitive reactions, *i.e.*, nitration, dimerization, *etc.*, with the participation of cation radicals[9] cannot be ruled out.

Methylation of compound **8** with methyl iodide in a KOH—acetone mixture afforded 1-(*N*-acetyl-*N*-methylamino)-4,5-bis(dimethylamino)naphthalene (**9**) in 91% yield. Acid hydrolysis of **9** resulted in a good yield of 4,5-bis(dimethylamino)-1-methylaminonaphthalene (**10**) which can easily undergo oxidation.

The exhaustive alkylation of naphthylamine **4** with methyl iodide in a KOH—DMF mixture gave the relatively stable tris-dimethylamino derivative **3** in 87% yield.

Comparing the ^{1}H NMR spectra of the synthesized amines (see Table 1), we noticed that the signals of the

Table 1. ^{1}H NMR spectra of the compounds synthesized ($CDCl_3$, δ, *J*/Hz)

Compound	H-3	H-2	H-8	H-7	H-6	4-NMe_2	5-NMe_2	Other groups
4	6.71 (d, $J_{2,3}$ = 8.06)	6.89 (m)	7.44 (dd, $J_{7,8}$ = 8.28)	7.33 (t, $J_{6,7}$ = 7.40)	7.03 (dd, $J_{6,8} \approx$ 1.1)	2.75 (s)	2.83 (s)	3.5 (br.s, NH_2, $^4J_{NH_2,H-2}$ = 2.56)
6	6.71 (d, $J_{2,3}$ = 7.91)	6.98 (d)	7.82 (dd, $J_{7,8}$ = 8.20)	7.48 (m, $J_{6,7}$ = 7.62)	7.48 (m, $J_{6,8}$ = 1.17)	2.81 (s)	—	3.3 (br.s, NH_2, $J_{5,7}$ = 1.47); 8.28 (dd, H-5, $J_{5,6}$ = 8.20)
7	6.90 (d, $J_{2,3}$ = 8.20)	7.09 (d)	8.08 (d, $J_{7,8}$ = 8.20)	7.38 (t, $J_{6,7}$ = 7.62)	6.99 (d)	2.84 (s)	2.80 (s)	8.12 (d, 2 H, H_b); 8.31 (d, 2 H, H_c); 8.59 (s, 1 H, H_a)
8a*	6.84 (d, $J_{2,3}$ = 8.20)	7.18 (d)	7.51 (d, $J_{7,8}$ = 8.20)	7.35 (m)	6.92 (m)	2.81 (s)	2.82 (s)	1.85 (s, COMe); 7.24 (br.s, NH)
8b*	6.92 (m)	7.35 (m)	7.48 (dd, $J_{7,8}$ = 8.20)	7.35 (m, $J_{6,7}$ = 7.62)	6.97 (dd, $J_{6,8} \approx$ 1.0)	2.78 (s)	2.80 (s)	2.27 (s, COMe); 7.07 (br.s, NH)
8**	6.89 (d, $J_{2,3}$ = 8.13)	7.30 (d)	7.45 (d, $J_{7,8}$ = 8.21)	7.30 (t, $J_{6,7}$ = 7.47)	6.94 (d)	2.72 (s)	2.73 (s)	2.10 (s, COMe); 9.55 (s, NH)
9**	6.91 (d, $J_{2,3}$ = 8.13)	7.27 (d)	7.10 (dd, $J_{7,8}$ = 8.20)	7.38 (t, $J_{6,7}$ = 7.51)	6.97 (dd, $J_{6,8}$ = 0.7)	2.74 (s)	2.75 (s)	1.57 (s, COMe); 3.13 (s, NMe)
10	6.54 (d, $J_{2,3}$ = 8.13)	6.91 (d)	7.37 (d, $J_{7,8}$ = 8.24)	7.29 (t, $J_{6,7}$ = 7.47)	6.95 (dd, $J_{6,8} \approx$ 1.1)	2.69 (s)	2.79 (s)	2.81 (s, NMe); 3.9 (br.s, NH)
3	6.86 (d, $J_{2,3}$ = 8.13)	6.97 (d)	7.85, (d, $J_{7,8}$ = 8.35)	7.30 (t, $J_{6,7}$ = 7.47)	6.90 (d, $J_{6,8}$ = 0.96)	2.78 (s)	2.79 (s)	2.72 (s, 1-NMe_2)

* The spectrum of a mixture of *Z*-*E*-isomers. ** In DMSO-d_6.

aromatic protons have a tendency to shift upfield in going from the "sesquisponge" **3** to compound **4** and then to the methylamino derivative **10**. Since in a series of similar compounds the degree of proton shielding usually changes in parallel with the electron donating effect of the substituents, one can conclude that in our case the +M-effect of the amino groups decreases in the $NHMe > NH_2 > NMe_2$ series. This does not fully agree with Hammett's constants for these substituents [σ_p −0.83 (NMe_2), −0.84 (NHMe), −0.66 (NH_2)], which can be explained by the steric interaction of the NMe_2 group with the H-8 proton. As shown previously,[10] this is the reason for the fact that in 1-aminonaphthalene the dihedral angle between the ring and the amino group plane is 28°.

The weakening of the influence of the 1-NMe_2 group in compound **3** also affects the difference in the chemical shifts of the 4-NMe_2 and 5-NMe_2 groups. This difference is only 0.007 ppm as compared with 0.077 and 0.097 ppm for amines **4** and **10**, respectively.

Taking the above into account, it was reasonable to expect that the basicity of the amino derivatives of the "proton sponge" would also change in the **10** > **4** > **3** series. We recently determined the basicity of compound **3** by potentiometric titration in acetonitrile: the pK_{a_1} and pK_{a_2} were found to be[1] 19.15 and 7.6. Unfortunately, it was rather difficult to carry out similar measurements for amines **4** and **10**, since they readily undergo oxidation. Therefore, by analogy with the "double proton sponge" **2** (see Ref. 5), we estimated[11] their first constants, pK_{a_1}, by the competitive method using ^{1}H NMR spectroscopy. We found that in an equimolar amount of **3** · $HClO_4$ and base **1** in DMSO-d_6, the first compound becomes 36% deprotonated at 20 °C. Taking into account the basicity constant of the "proton sponge" in DMSO, these measurement give pK_{a_1} = 8.0 for amine **3**. Under similar conditions, **5** · $HClO_4$ is 11% deprotonated by "sesquisponge" **3**, and perchlorate **10** · $HClO_4$ is 8.5% deprotonated. Thus, the calculated basicities of these compounds in DMSO are pK_{a_1}(**5**) = 9.8 and pK_{a_1}(**10**) = 10.1. The latter ionization constant indicates that the methylamino derivative of the "proton sponge" **10** is a stronger base than compound **2**. To put it another way, the +M-effect of the one NHMe group in compound **10** is larger than the donor effect of the two sterically interacting *peri*-NMe_2-groups in amine **2**.

It is noteworthy that the salt of amino derivative **4** does not undergo deprotonation by the "proton sponge" even after prolonged heating (45 °C, 24 h), which may be due to the large difference in the basicities of compounds **1** and **4** (2.3 pK_a units).

An attempt to estimate the pK_{a_1} of the synthesized amines in acetonitrile by the competitive method was unsuccessful. The ^{1}H NMR spectra of similar mixtures of a cation and a base at ~20 °C in CD_3CN were composed of wide unresolved peaks indicating strong inhibition of proton exchange. Taking into account the low nucleophilicity of "proton sponges", it is very unlikely that the transfer of protons between "proton sponge" cations and bases occurs through immediate contact. Obviously, this requires small and sterically un-hindered transferring molecules with rather high basicity. Dimethyl sulfoxide (pK_a = 0) can play such a role but acetonitrile, whose basicity is 10 orders of magnitude lower (pK_a = −10.1)[12] cannot (*cf.* the data on deprotonation of the cation of the nitro derivative **5** in DMSO and in other solvents).[13]

Experimental

^{1}H NMR spectra were obtained on a Unity-300 instrument (300 MHz) with $SiMe_4$ as the internal standard. Before recording the spectra, solutions of equimolar mixtures of perchlorates and bases in DMSO-d_6 (the concentration of each component was $4 \cdot 10^{-2}$ mol L^{-1}) were kept at 45 °C for 1 h. IR spectra were recorded on an IKS-40 instrument. The UV spectrum was obtained on a Specord M-40 spectrometer. Hydrogenation was carried out using a WU-4 shaker (120—150 rpm). Hydrogen was obtained by the reaction of 20% KOH with granulated aluminum. To remove moisture, the catalyst (2% Pd/C) was evacuated at 2 Torr for 1 day. Chromatography was carried out on Al_2O_3 with degree V of Brockmann activity. Melting points were determined in sealed capillaries on a PTP instrument and were not corrected.

1-Amino-4,5-bis(dimethylamino)naphthalene (4). ***A.*** The catalyst (0.25 g) was placed in a solution of compound **5** (0.26 g, 1 mmol) in EtOH (70 mL). The reaction vessel was flushed with hydrogen, and hydrogenation was carried out at ~20 °C and under H_2 at atmospheric pressure. After 5 to 5.5 h, the solution was quickly filtered, and the filtrate was concentrated on a rotary evaporator at 40—45 °C. The yield was 0.23 g (100%).

B. Hydrogenation of compound **5** in EtOAc was carried out at 70 °C for 8 h. The yield was 100 %.

Amine **4** is yellowish oil that rapidly darkens in air and under light. Its perchlorate formed as light gray crystals, m.p. 256—258 °C (dec., from EtOH). Found (%): C, 50.82; H, 6.15; Cl, 10.64; N, 12.71. $C_{14}H_{20}ClN_3O_4$. Calculated (%): C, 50.99; H, 6.11; Cl, 10.75; N, 12.74.

***N,N*-Dimethyl-1,4-naphthylenediamine (6).** The reduction of nitronaphthylamine **5** in EtOH was carried out for 30 h. The reaction product was isolated by a procedure similar to that described above. The yield was quantitative (0.19 g). Naphthylenediamine **6** is a light yellow liquid, readily oxidizable in air.[14] IR (film), ν/cm^{-1}: 1589 (arom.); 1624, 2782 (Me); 3235, 3343 (NH_2).

1-(*p*-Nitrobenzilideneamino)-4,5-bis(dimethylamino) naphthalene (7). A mixture of amine **4** (0.23 g, 1 mmol) and *p*-nitrobenzaldehyde (0.15 g, 1 mmol) in EtOH (70 mL) was refluxed for 2 h and then concentrated on a water bath. The residue was chromatographed on Al_2O_3 (d = 4 cm, l = 30 cm, the eluent was $CHCl_3$). The dark red fraction with R_f 0.83 was collected to afford 0.29 g (81 %) of azomethine **7** as wine-colored crystals, m.p. 148—149 °C (from EtOH). Found (%): C, 69.52; H, 6.21; N, 15.42. $C_{21}H_{22}N_4O_2$. Calculated (%): C, 69.59; H, 6.12; N, 15.46. UV (MeOH), λ_{max}/nm: 284 (4.20), 363 (3.96), 490 (sh, 3.01).

1-Acetamido-4,5-bis(dimethylamino)naphthalene (8). Ac_2O (0.189 mL, 2 mmol) was added to a solution of amine **4** (0.46 g, 2 mmol) in EtOAc (100 mL) prepared according to procedure ***B***. The mixture was stirred at ~20 °C for 20 h and

then evaporated on a water bath. The residue was triturated with 25% ammonia (10 mL), extracted with $CHCl_3$ (3 × 10 mL), and chromatographed on an Al_2O_3 column (d = 2 cm, l = 27 cm, the eluent was $CHCl_3$). The light lilac fraction with R_f 0.40 was collected to yield 0.41 g (76%) of amide **8** as light lilac leaflets, m.p. 124—125 °C (from a Pr^iOH—H_2O mixture, 1 : 3). Found (%): C, 71.05; H, 7.84; N, 15.51. $C_{16}H_{21}N_3O$. Calculated (%): C, 70.82; H, 7.80; N, 15.48. IR (Vaseline oil), ν/cm^{-1}: 1548, 1576 (arom.); 1650 (C=O); 3233 (NH).

Diazotization of 1-amino-4,5-bis(dimethylamino) naphthalene (5) and Sandmeyer's reaction. Concentrated HBr (0.16 mL) was added to a solution of amine **4** (0.23 g, 1 mmol) in EtOH (70 mL) prepared according to the procedure *A*, and the mixture was cooled to 0 °C. A solution of $NaNO_2$ (0.081 g, 1.2 mmol) in H_2O (2 mL) was added dropwise with vigorous stirring, then a solution of Cu_2Br_2 (0.15 g, 1 mmol) in 40% HBr (1 mL) was added, and the mixture was concentrated to 1/4 of its volume. The mixture was then cooled and alkalized with 10% KOH to pH 9.0, and the products were extracted with hexane (4 × 3 mL). Chromatography on Al_2O_3 allowed us to separate the product from resins and other admixtures to give 0.073 g (25%) of 1-bromo-4,5-bis(dimethylamino)-naphthalene as a pale yellow oil, R_f 0.78 (see Ref. 15).

1-(*N*-Acetyl-*N*-methylamino)-4,5-bis(dimethylamino)-naphthalene (9). KOH powder (0.030 g, 0.53 mmol) was added to a solution of amide **8** (0.136 g, 0.5 mmol) in acetone (6 mL), and then MeI (0.100 mL, 1.6 mmol) was added. The mixture was stirred at ~20 °C for 4 days, the acetone was evaporated, the residue was extracted with $CHCl_3$, and the product was chromatographed on an Al_2O_3 column (d = 1.7 cm, l = 25 cm, $CHCl_3$ as the eluent). The fraction with R_f 0.67 (TLC control) was collected to give 0.13 g (91%) of amide **9** as light cream-colored crystals, m.p. 116—118 °C (from *n*-octane). Found (%): C, 71.58; H, 8.17; N, 14.69. $C_{17}H_{23}N_3O$. Calculated (%): C, 71.55; H, 8.12; N, 14.72.

4,5-Bis(dimethylamino)-1-methylaminonaphthalene (10). A solution of amide **9** (0.082 g) in concentrated HCl (5 mL) was refluxed for 7 h, and the mixture was then treated with 20% KOH (15 mL). Amine **10** formed and was extracted with benzene (3×3 mL). The solvent was distilled off to yield 0.065 g (93%) of a light yellow oil rapidly darkening in the air, R_f 0.20. Perchlorate: colorless crystals, m.p. 245—247 °C (dec., from EtOH). Found (%): C, 52.34; H, 6.49; Cl, 10.27; N, 12.23. $C_{15}H_{22}ClN_3O_4$. Calculated (%): C, 52.40; H, 6.45; Cl, 10.31; N, 12.22.

1,4,5-Tris(dimethylamino)naphthalene (3). KOH powder (1.0 g, 8.6 mmol) was added to a solution of amine **4** (0.46 g, 2 mmol) synthesized by procedure *B*, and the mixture was stirred in an inert atmosphere for 5 min. After that, MeI (1.0 mL, 8 mmol) was added, and the mixture was stirred at ~20 °C for 1 h and at 100 °C for 1 h. The mixture was diluted with ice water (100 mL), alkalized with 25% ammonia (10 mL), and extracted with benzene (4×20 mL). The solvent was evaporated, and the residue was evacuated (2 Torr) to yield 0.45 g (87%) of naphthylamine **3** as a reddish oil, which oxidizes relatively slowly in the air, R_f 0.87 (in an 1 : 3 AcOEt—C_6H_6 mixture) or 0.26 ($CHCl_3$). Found (%): C, 74.72; H, 9.05; N, 16.30. $C_{16}H_{23}N_3$. Calculated (%): C, 74.66; H, 9.01; N, 16.33. IR (in a thin film), ν/cm^{-1}: 1580 (arom.); 2790 (Me). ^{13}C NMR (75.4 MHz, $CDCl_3$), δ: 44.7 (q, 4- and 5-NMe_2, ^1J = 133.2 Hz); 45.7 (q, 1-NMe_2, ^1J = 133.4 Hz); 112.6 (d, C-3, C-6, ^1J = 155.7 Hz); 114.2 (d, C-8, ^1J = 155.5 Hz); 117.4 (d, C-2, ^1J = 161.4 Hz); 121.9 (s, C-4a); 124.9 (d, C-7, ^1J = 157.2 Hz); 133.3 (s, C-8a); 145.3 (s, C-1); 146.7 (s, C-4); 150.9 (s, C-5). Perchlorate, colorless crystals, m.p. 206—208 °C (dec., from EtOH). Found (%): C, 54.00; H, 6.82; Cl, 9.85; N, 11.72. $C_{16}H_{24}ClN_3O_4$. Calculated (%): C, 53.71; H, 6.76; Cl, 9.91; N, 11.74.

This study was financially supported by the Russian Foundation for Basic Research (Project No. 96-03-32153a).

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Received February 4, 1997