peri-Naphthylenediamines
33.* Further studies of the reactions of 1,8-bis(dimethylamino)naphthalene with trifluoroacetic anhydride

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The reaction of 1,8-bis(dimethylamino)naphthalene (**1**) ("proton sponge") with a large excess of trifluoroacetic anhydride in the absence of a solvent afforded a complex mixture of products among which were *trans*- (**2**) and *cis*-diols (**3**) of the naphtho[1,8-*c*,*d*]pyran series, the double "proton sponge" (**4**), its di- (**5**) and tetrafluoroacetyl (**6**) derivatives, and compound **7**. These results differ substantially from those obtained previously in the reactions performed in various solvents. Some conversions of compounds **4** and **7** were investigated. The ^{19}F NMR spectra of the compounds synthesized are discussed.

Key words: 1,8-bis(dimethylamino)naphthalene, "proton sponge," trifluoroacetic anhydride, electrophilic substitution, ^{1}H and ^{19}F NMR spectra.

Recently,[2] we have discovered a new reaction involving compounds of the naphthalene series; *i. e.*, the reaction of trifluoroacetic anhydride (TFAA) with 1,8-bis(dimethylamino)naphthalene (**1**) ("proton sponge") in various solvents afforded predominantly naphtho[1,8-*c*,*d*]pyran derivatives **2**—**4**. The yield of double "proton sponge" **4** reached 65%, whereas diols **2** and **3** were obtained in 7.5 and 3.7% yields, respectively. The present work was undertaken as part of our continuing studies of these reactions.

Results and Discussion

Previously,[2] it has been found that the results of trifluoroacetylation of compound **1** depend substantially on the solvent, temperature, and the reaction time. In all experiments performed, an amount of trifluoroacetic anhydride (TFAA) used did not exceed 1.5 mole per mole of diamine **1**. This raised the question of whether the ratio between the reaction products would be changed in the presence of a large excess of TFAA, the latter being used both as the reagent and the solvent. In particular, we expected that the reactions performed under these conditions would afford diols **2** and **3** in substantially higher yields.

Actually, the yields of diols **2** and **3** were increased by a factor of more than two (16 and 10%, respectively) upon stirring of compound **1** with a tenfold excess of TFAA at ~20 °C, while the yield of dimer **4** was decreased to 5%. Simultaneously, bis- and tetrakis-trifluoroacetylation products of the latter (**5** and **6**, respectively) were isolated from the reaction mixture in 6 and 3% yields, respectively. In addition, a chromatographically mobile yellow compound was obtained in 15% yield to which structure **7** was assigned (Scheme 1).

The structures of the previously unknown derivatives **5**—**7** were confirmed by ^{1}H and ^{19}F NMR spectroscopy. The structure of compound **7** was supported by the data from mass spectrometry (unfortunately, we failed to grow crystals of compound **7** suitable for X-ray diffraction analysis). The ^{19}F NMR spectrum of compound **5** has only two singlets belonging to pairs of the equivalent CF_3 and $COCF_3$ groups. Evidently, if the $COCF_3$ groups in the compound had the *cis* arrangement (isomer **8**), its ^{1}H NMR spectrum would be the same, but its ^{19}F NMR spectrum would have three signals due to the nonequivalence of the CF_3 groups.

The ^{1}H and ^{19}F NMR spectral data for compound **5** are also consistent with isomeric *trans*-structure **9**, which seems at first glance to be even more probable. The fact that only monohydrochloride was formed when dry HCl was passed through a solution of compound **5** in CH_2Cl_2 is the major argument in favor of its *cis*-structure. Taking into account that the basicity of tetraacylated product **6** is so much lower that it does not form salts at all under the action of strong acids, the resulting cation has, apparently, structure **10a**.

Interestingly, the intramolecular hydrogen bond in cation **10a**, like that in monocation **10b** of the unsubstituted compound, is somewhat asymmetric and is characterized by the spin-spin coupling constants between the chelated proton and two nonequivalent

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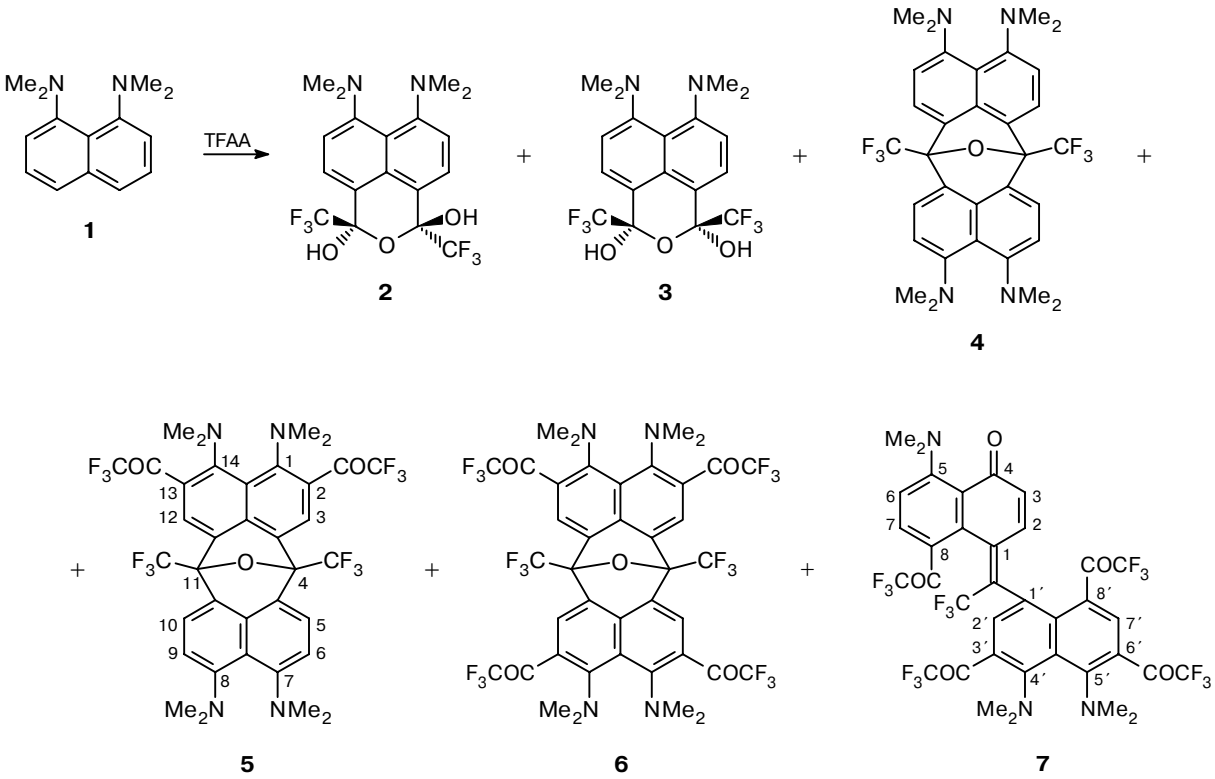
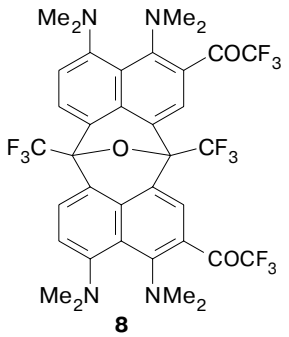
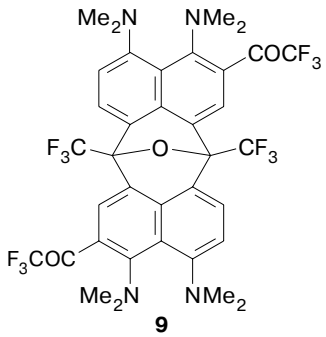
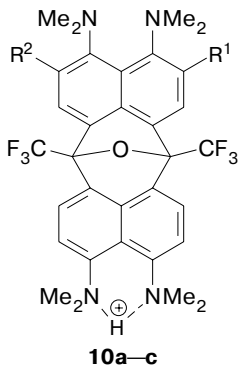
Scheme 1



NMe_2 groups equal to 2.11 and 1.99 Hz (for **10b**, 1.98 and 1.87 Hz, respectively).[2] Undoubtedly, compound **9** could interact with acids to form dications with strongly asymmetric intramolecular hydrogen bonds (*cf.* Ref. 3).







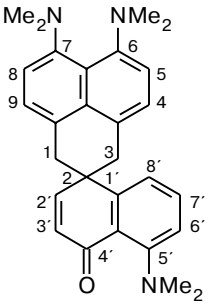
	R^1	R^2
10		
a	$COCF_3$	$COCF_3$
b	H	H
c	$COCF_3$	H

The fact that the ^{1}H NMR spectrum of compound **7** has two doublets for the H(2) and H(3) protons with the characteristic quinoid *ortho*-spin-spin coupling constant (J_o = 10.3 Hz) (*cf.* the data for ketone **11**,[4] which was used as the reference compound) is the major argument in support of the quinoid nature of this compound. In addition, the NMR spectrum of derivative **7** has signals of only three dimethylamino groups, which indicate that dimerization of two residues of compound **1** was accompanied by hydrolytic replacement of one NMe_2 group by the carbonyl fragment. This process has been repeatedly observed for different compounds.[4,5]

Yet another characteristic feature of the ^{1}H NMR spectrum of compound **7** is associated with the signals for the H(2′) and H(7′) protons at δ 8.16 and 8.36, respectively, which are substantially broadened due to spin-spin coupling with the fluorine atoms of two nonequivalent CF_3 groups. The low-field signal was assigned to the H(7′) proton based on the fact that its chemical shift is similar to that of the H(3) proton in diketone **12**[2] characterized by a similar environment (δ 8.48). Analogous spin-spin coupling with the 4-$COCF_3$ group is also characteristic of the H(7) atom, which is manifested as a doublet of quartets at δ 7.50 ($^5J_{H,F}$ = 1.86 Hz, $^3J_{7,6}$ = 8.90 Hz). Correspondingly, the signal for the *ortho*-H(6) proton is observed as a doublet at δ 7.08 with $^3J_{6,7}$ = 8.90 Hz. Theoretically, one of two *ortho*-$COCF_3$ groups can be located at position 6 of compound **7**, the overall ^{1}H NMR spectral pattern being the same.

We believe that the latter situation is less probable for reasons associated with the assumed chemistry of formation of compound **7** (see below) involving the immonium group as a precursor of the quinoid carbonyl fragment. The former should passivate substantially the benzene ring adjacent to the quinoid group with respect to electrophilic substitution.

The assignment of five signals of the fluorine-containing groups in the ^{19}F NMR spectrum of compound **7** was made based on a comparison of this spectrum with the spectra of compounds **4**—**6**, monoketone **13**,[2] and diketone **12** (Table 1). The CF_3 groups, which do not belong to the acyl group, can be readily identified because their signals are observed at substantially higher field compared to the signals of the $COCF_3$ groups. At the same time, it is rather difficult to make the precise assignment of the signals of these groups due to a rather small difference in their chemical shifts. Thus, the positions of the signals of the 2-$COCF_3$ and 4-$COCF_3$ groups in diketone **12** differ by only 0.6 ppm, while the spin-spin coupling constants with the H(3) proton are virtually identical ($^5J_{F,H} \approx 1.5$ Hz). However, the signals of the *ortho*-$COCF_3$ groups in compounds **5** and **6** are observed at somewhat higher field than the signal of the *para*-$COCF_3$ group in ketone **13**. We are guided by this tendency in the assignment of the signals in the spectra of the other compounds. It is needless to say that the assignment of the signals of two $COCF_3$ groups at the *ortho*-(3′- and 6′-) or *para*-(8- and 8′-) positions in compound **7** is arbitrary.



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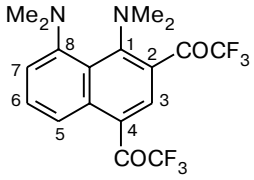

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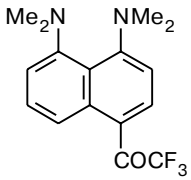

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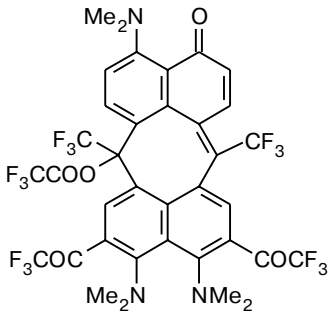


14

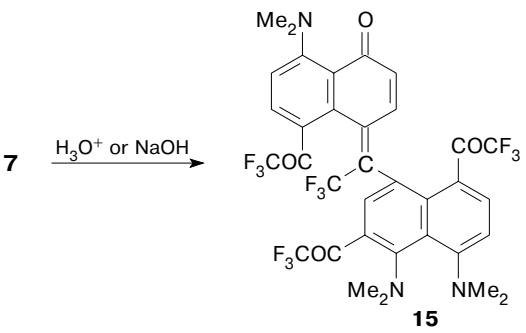
When establishing the structure of compound **7**, we initially assumed that it may be an *O*-trifluoroacetyl derivative of diol or intermediate alcohol formed in the course of preparation of dimer **4**. In particular, we considered structure **14** as a possibility. Since the trifluoroacetoxy group would be expected to be readily hydrolyzed, we heated compound **7** both in acidic and alkaline media. It appeared that hydrolytic decomposition of compound **7** proceeded rather difficultly (prolonged boiling was required), in both cases the same product which did not contain the hydroxyl group being formed. The molecular weight of this product (*m/z* is 767) corresponds to the weight of compound **7** minus one trifluoroacetyl group.

Table 1. Chemical shifts in the ^{19}F NMR spectra ($CDCl_3$, δ) of compounds **4**—**7**, **12**, **13**, and **15**

Compound	*ortho*-$COCF_3$	*para*-$COCF_3$	CF_3
4	—	—	−74.02
5	−69.64	—	−74.61
6	−70.01	—	−75.38
7	−69.81 (6′-$COCF_3$) −69.58 (3′-$COCF_3$)	−67.05 (8-$COCF_3$) −66.83 (8′-$COCF_3$)	−79.00
12	−69.84	−69.26	—
13	—	−68.97	—
15	−69.45 (3′-$COCF_3$)	−67.03 (8-$COCF_3$) −66.37 (8′-$COCF_3$)	−79.00

Apparently, difficulties associated with hydrolysis of compound **7** are indicative of the absence of the trifluoracetoxy group in the starting compound. Assuming that structure **7** is correct and based on the data of Table 1, it can be concluded that one of two *ortho*-$COCF_3$ groups was eliminated. The ^1H NMR spectrum of the resulting compound is best consistent with elimination of the 6′-$COCF_3$ group, *i.e.*, with structure **15** (Scheme 2).

Scheme 2



15

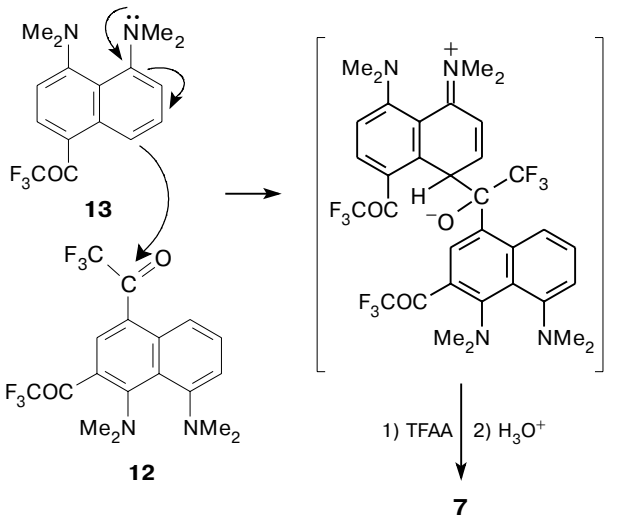
In the ^1H NMR spectrum of compound **15**, two new signals, *viz.*, a doublet for the H(6′) proton and a doublet of quartets for the H(7′) proton, appear instead of a single signal for the H(7′) proton. It can also be seen from Table 1 that the position of the signal of one *para*-$COCF_3$ group remains virtually unchanged on going from compound **7** to compound **15** (δ −67.05 and −67.03, respectively), whereas the second signal is slightly shifted downfield (from δ −66.83 to −66.37). It is reasonable to assume that compounds **7** and **15** bear the 8-$COCF_3$ and 8′-$COCF_3$ groups, respectively.

Apparently, hydrolysis of compound **7** in an acidic medium proceeded according to the mechanism of usual electrophilic protodeacylation, whereas hydrolysis in an alkaline medium occurred, most likely, as the haloformic

cleavage with the intermediate formation of the corresponding carboxylate anion (*cf.* Ref. 6) followed by its carboxylation. In this connection, noteworthy is the surprisingly high regioselectivity and the same direction of the process in acidic and alkaline media.

Formally, the formation of compound **7** can be represented as a result of nucleophilic addition of monoketone **13** to the carbonyl group of diketone **12** followed by elimination of the water molecule, repeated acylation, and hydrolytic replacement of the dimethylammonium group by the carbonyl fragment (Scheme 3).

Scheme 3



This sequence of conversions agrees with the known high CH-nucleophilicity of diamine **1**.[7,8] In addition, it was found that dimer **4** was formed from two molecules of monoketone **13** in a similar way.[2] The fact that diamine **1** rather than monoketone **13** is involved in the initial stage of the reaction must not be ruled out. It is less probable that intermediate tri- or tetracarbonyl derivatives of compound **1** enter into the reaction instead of diketone. We observed no evidence of formation of the latter in numerous experiments on trifluoroacetylation of compound **1**.

It was of interest to study trifluoroacetylation of compound **4**. It appeared that the reaction of equimolar amounts of dimer **4** and TFAA or the reaction even in the presence of a fourfold excess of TFAA in both CH_2Cl_2 and dichloroethane did not proceeded with a noticeable rate. The reaction started only in the presence of a large excess of TFAA (a tenfold excess is optimum) in the absence of a solvent to give diketone **5** and tetraketone **6** in 5 and 8% yields, respectively (the mixture was kept at ~20 °C for 48 h), mono- and triketones being absent.

It was established that trifluoroacetylation of naphthopyran **4** afforded compounds containing only even numbers of $COCF_3$ groups, which is, apparently, associated with the fact that "proton sponges" are involved in these reactions predominantly in the nonprotonated form. This is supported by the fact that the reaction of dimer **4** with a large excess of TFAA at high temperature (refluxing for 5 h) did not lead to an increase in the yields of derivatives **5** and **6**. Apparently, the primary reaction product was converted into monocation **10c** under the action of the acid eliminated after insertion of the first $COCF_3$ group into molecule **4**. Evidently, the naphthalene fragment, which already contains the $COCF_3$ group, *i.e.*, which possesses lower basicity and hence can exist, at least partially, in the nonprotonated form, is more active with respect to the subsequent replacement. Then trifluoroacetylation of the second naphthalene fragment proceeds analogously. It should be taken into account that the insertion of each successive $COCF_3$ group leads to a decrease in basicity of the substitution product formed and to an increase in the concentration of the free base. Hence, the subsequent stages of the reaction should proceed more readily than the preceding stages. This resembles nitration of 1,8-bis(dimethylamino)naphthalene (**1**) with the HNO_3—AcOH system affording only the 2,4,5,7-tetranitro derivative even in the case of deficiency of nitric acid.[9]

In conclusion it should be emphasized that trifluoroacetylation of compound **4** is the first example of electrophilic substitution in double "proton sponges." The regularities found in the present study would be expected to be common to other analogous substrates and electrophiles.

Experimental

The ^1H and ^{19}F NMR spectra were measured on a Unity-300 instrument (300 MHz) with $SiMe_4$ and CCl_3F as the internal standards, respectively. The IR spectra were recorded on a Specord IR-75 spectrometer. The mass spectra were obtained on a Kratos-MF-30 instrument. Cromatography was carried out with the use of Al_2O_3 (Brockmann II); the eluents are mentioned where appropriate in the Experimetnal.

The reagents and solvents were purified and dried according to standard procedures.[10]

Trifluoroacetylation of diamine 1 with an excess of TFAA. Trifluoroacetic anhydride (6 mL) was added dropwise with stirring to compound **1** (1 g, 4.7 mmol) at −15 °C for 20 min. Then the reaction mixture was stirred at −15 °C for 30 min, kept at 20 °C for 48 h, concentrated to dryness, and suspended in CH_2Cl_2 (20 mL). The white precipitate that formed was filtered off and washed with CH_2Cl_2 (3×2 mL). Then the precipitate and the filtrate were worked up separately.

The precipitate was stirred in Me_2CO (30 mL) for 1 h after which the white heavy precipitate that remained undissolved was filtered off and washed with Me_2CO. ***trans*-Pyran trifluoroacetate** (**2**) was obtained in a yield of 0.4 g (16%). Then Me_2CO was distilled off from the filtrate and the residue was recrystallized from MeCN to obtain ***cis*-pyran trifluoroacetate** (**3**) in a yield of 0.25 g (10%). Treatment of the resulting salts with an aqueous solution of NaOH afforded bases **2** and **3** whose properties are identical with those of the specimens obtained previously.[2]

Dichloromethane was distilled off from the filtrate, the residue was suspended in H_2O (15 mL), the mixture was stirred for 30 min, and the orange precipitate that formed was filtered off. The aqueous solution was alkalized with a 10% NaOH solution to pH 14 and extracted with $CHCl_3$ (5×5 mL). The

extract was concentrated to a minimum volume and the residue was chromatographed on a column (h = 20 cm, d = 2.5 cm) using $CHCl_3$ as the eluent. Compound **4** (R_f 0.82) and the starting compound **1** (R_f 0.47) were isolated in yields of 0.07 g (5%) and 0.3 g (30%), respectively.

The orange precipitate that was filtered off from the aqueous solution was suspended in water (2 mL), alkalized with a 10% NaOH solution to pH 14, and extracted with $CHCl_3$ (5×5 mL). After removal of the solvent, the residue was chromatograhed on a column (h = 20 cm, d = 2.5 cm) using benzene as the eluent. Diketone **1,7,8,14-tetrakis(dimethylamino)-2,13-bis(trifluoroacetyl)-4,11-bis(trifluoromethyl)-4,11-epoxydinaphtho[1,8-*a*,*b*;1′,8′-*e*,*f*]cyclooctane (5)** (R_f 0.76), tetraketone **1,7,8,14-tetrakis(dimethylamino)-2,6,9,13-tetrakis(trifluoroacetyl)-4,11-bis(trifluoromethyl)-4,11-epoxydinaphtho[1,8-*a*,*b*;1′,8′-*e*,*f*]cyclooctane (6)** (R_f 0.38), and **5-dimethylamino-4-oxo-8-trifluoroacetyl-1-{1-[4,5-bis(dimethylamino)-3,6,8-tris(trifluoroacetyl)-1-naphthyl]-2,2,2-trifluoroethylidene}-1,4-dihydronaphthalene (7)** (R_f 0.10) were isolated in yields of 0.11 g (6%), 0.06 g (3%), and 0.3 g (15%), respectively. Compound **5** was obtained as orange crystals, m.p. 270—272 °C, R_f 0.55 (Al_2O_3, $CHCl_3$). Found (%): C, 54.44; H, 3.80; F, 28.72; N, 7.08. $C_{36}H_{30}F_{12}N_4O_3$. Calculated (%): C, 54.41; H, 3.78; F, 28.71; N, 7.05. ^{1}H NMR (25 °C, $CDCl_3$), δ: 2.75 (s, 12 H, C(7)NMe_2, C(8)NMe_2); 2.92 (s, 12 H, C(1)NMe_2, C(14)NMe_2); 6.85 (d, 2 H, H(6), H(9), J_o = 8.3 Hz); 7.59 (br.d, 2 H, H(5), H(10)); 7.96 (s, 2 H, H(3), H(12)).

1,8,14-Tris(dimethylamino)-7-dimethylammonio-2,13-bis(trifluorolacetyl)-4,11-bis(trifluoromethyl)-4,11-epoxydinaphtho[1,8-*a*,*b*;1′,8′-e,f]cyclooctane chloride (10a) was obtained in quantitative yield as yellow crystals by passing dry HCl through a solution of compound **5** in CH_2Cl_2, m.p. 247—249 °C (with decomp.). Compound **10a** was hydrolyzed upon heating in water to form base **5**. ^{1}H NMR (25 °C, CD_3CN), δ: 2.90 (br.s, 12 H, C(1)NMe_2, C(14)NMe_2); 3.06 (d, 6 H, C(7)NMe_2 or C(8)NMe_2, $J_{NMe2,H}$ = 2.1 Hz); 3.11 (d, 6 H, C(8)NMe_2 or C(7)NMe_2, $J_{NMe2,H}$ = 2.0 Hz); 7.95 (m, 4 H, H(5), H(6), H(9), H(10)); 8.05 (m, 2 H, H(3), H(12)); 18.66 (br.s, 1 H, NH).

Compound **6** was obtained as orange crystals, m.p. >300 °C, R_f 0.31 (Al_2O_3, $CHCl_3$). Found (%): C, 48.70; H, 2.89; F, 34.72; N, 5.62. $C_{40}H_{28}F_{18}N_4O_5$. Calculated (%): C, 48.68; H, 2.84; F, 34.68; N, 5.68. IR (Nujol mulls), ν/cm^{-1}: 1686 (C=O); 1580, 1513 (ring). ^{1}H NMR (25 °C, $CDCl_3$), δ: 2.94 (s, 24 H, 4 NMe_2); 7.97 (s, 4 H, H(3), H(5), H(10), H(12)). Compound **7** was obtained as yellow crystals, m.p. 240—242 °C (from *n*-octane). Found (%): C, 50.18; H, 2.89; F, 33.06; N, 4.80. $C_{36}H_{24}F_{15}N_3O_5$. Calculated (%): C, 50.05; H, 2.78; F, 33.02; N, 4.86. ^{1}H NMR (25 °C, $CDCl_3$), δ: 2.98 (s, 6 H, C(5)NMe_2); 3.02 (br.s, 12 H, 2 NMe_2); 6.24 (d, 1 H, H(3), $J_{3,2}$ = 10.3 Hz); 6.50 (d, 1 H, H(2), $J_{2,3}$ = 10.3 Hz); 7.08 (d, 1 H, H(6), $J_{6,7}$ = 8.9 Hz); 7.50 (dq, 1 H, H(7), $J_{7,6}$ = 8.8 Hz, $J_{H,F}$ = 1.9 Hz); 8.16 (br.s, 1 H, H(2′)); 8.36 (br.s, 1 H, H(7′)).

5-Dimethylamino-4-oxo-8-trifluoroacetyl-1-{1-[4,5-bis(dimethylamino)-3,8-bis(trifluoroacetyl)-1-naphthyl]-2,2,2-trifluoroethylidene}-1,4-dihydronaphthalene (15). Compound **7** (0.1 g, 0.12 mmol) was dissolved in a minimum amount of Me_2CO and then a 20% NaOH solution (10 mL) was added. The reaction mixture was refluxed with stirring for 3 h and extracted with $CHCl_3$. The solvent was removed and the residue was chromatographed on a column (h = 15 cm, d = 1 cm) using $CHCl_3$ as the eluent. The starting compound **7** (R_f 0.50) was isolated in a yield of 0.05 g (50%). Compound **12** (R_f = 0.60) was obtained as yellow crystals in a yield of 0.44 g (50%), m.p. 114—116 °C (from *n*-octane). Found (%): C, 53.25; H, 3.31; F, 29.76; N, 5.51. $C_{34}H_{25}F_{12}N_3O_4$. Calculated (%): C, 53.19; H, 3.26; F, 29.73; N, 5.47. ^{1}H NMR (25 °C, $CDCl_3$), δ: 2.93 (s, 6 H, C(5′)NMe_2); 2.98 (s, 6 H, C(5)NMe_2); 3.21 (s, 6 H, C(4′)NMe_2); 6.24 (d, 1 H, H(3), $J_{3,2}$ = 10.3 Hz); 6.48 (d, 1 H, H(2), $J_{2,3}$ = 10.3 Hz); 6.86 (d, 1 H, H(6′), $J_{6',7'}$ = 8.9 Hz); 7.06 (d, 1 H, H(6), $J_{6,7}$ = 8.9 Hz); 7.48 (dq, 1 H, H(7), $J_{7,6}$ = 8.8 Hz, $J_{H,F}$ = 1.8 Hz); 7.81 (dq, 1 H, H(7′), $J_{7',6'}$ = 8.9 Hz, $J_{H,F}$ = 1.5 Hz); 7.96 (s, 1 H, H(2′)).

Trifluoroacetylation of compound 4 in an excess of TFAA. Trifluoroacetic anhydride (0.5 mL) was added dropwise with stirring to compound **4** (0.22 g, 0.36 mmol) at −15 °C. The reaction mixture was stirred at −15 °C for 30 min, kept at 20 °C for 48 h, and concentrated to dryness in air. The orange residue was suspended in CH_2Cl_2. The white precipitate that formed was filtered off and washed several times with CH_2Cl_2. Trifluoroacetate of the starting compound **4** was obtained in a yield of 0.23 g. The filtrate was concentrated and chromatographed on a column (h = 15 cm, d = 1 cm) using benzene as the eluent. Compounds **5** (R_f 0.76) and **6** (R_f = 0.38) were isolated in yields of 0.015 g (5%) and 0.03 g (8%), respectively.

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