

Reactions of resorcinarene with *N,N*-dimethylcarbamoyl and *N,N*-dimethylthiocarbamoyl chlorides: factors affecting the process performance

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Abstract

Carbamoylation and thiocarbamoylation of resorcinarenes in the presence of alkali metal carbonates were studied. The effects of the pre-organization of the resorcinarene molecule, the base used, and the nature of the acylating reagent on the reaction outcome were demonstrated. It was shown that octacarbamoylated resorcinarenes in the *boat* conformation show high absorption properties and selectivity towards cesium cations.

Keywords: Resorcinarenes, pre-organization, carbamoylation, thiocarbamoylation, alkali metal cations, complexation

Introduction

Resorcinarenes, polyhydroxyaromatic compounds having a molecular cavity, form a convenient basis for the design of receptor systems, because of the ease of modification of these compounds and the possibility of immobilization of the macrocycle in a definite conformation.¹⁻⁴ O-Acylation⁵⁻⁹ and O-alkylation of resorcinarenes¹⁰⁻¹⁵ are used most often to introduce ionophoric groups. Using these reactions, a broad range of polyfunctionalized derivatives of resorcinarenes have been synthesized; many of these compounds exhibit acceptor properties towards metal cations.¹⁶⁻²¹ It is noteworthy that the receptor properties of the synthesized compounds and their selectivity are determined by pre-organization of the macrocyclic core of resorcinarene and the nature of the immobilized ionophoric groups.

In order to create new receptor systems with functional groups containing various sets of electron-donating atoms and located in space in a definite way, we studied carbamoylation and thiocarbamoylation of resorcinarenes with a specific pre-organization of the macrocyclic core.

In agreement with this goal, as macrocyclic substrates, we chose resorcinarenes **1** with *rccc* configuration of alkyl (**a-d**), alkylaryl (**e**) and phenyl (**f**) substituents in the methylenedioxy bridges, for which *crown* is the major conformation, and tetranaphthylresorcinarene **2**, which occurs in the *chair* conformation with the *rctt* configuration of the naphthyl moieties (Fig. 1). *N,N*-Dimethylcarbamoyl and *N,N*-dimethylthiocarbamoyl chlorides served as the acylating reagents.

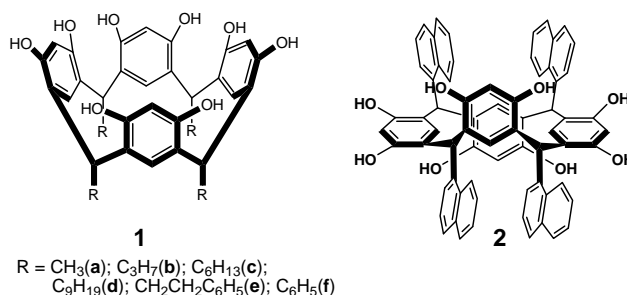


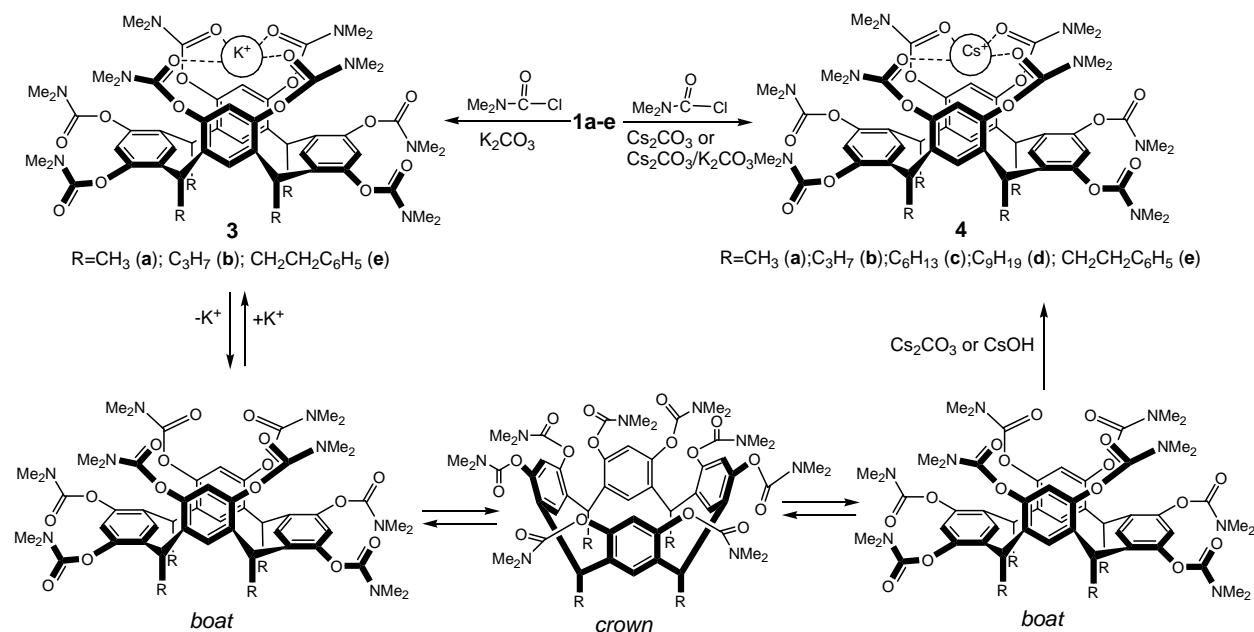
Figure 1. Structure of resorcinarenes **1,2**.

Results and Discussion

Carbamoylation of resorcinarenes **1** and **2** with *N,N*-dimethylcarbamoyl chloride was carried out in acetone at 55-60 °C. Alkali metal carbonates (Na₂CO₃, K₂CO₃, Cs₂CO₃) were used as bases. The resorcinarene : carbamoyl chloride : carbonate reactant ratio was 1:12:12.

In the presence of Na₂CO₃, the reaction was in all cases slow and non-selective. Even long-term heating (for more than 40 h) of the reaction mixtures did not result in the formation of percarbamoylated resorcinarenes, as indicated by MALDI data for the obtained products.

The results of carbamoylation of resorcinarenes **1** and **2** in the presence of potassium and cesium carbonate depended on the pre-organization of the macrocyclic substrate and the nature of the carbonate. When *rccc* resorcinarenes **1a,b,e** reacted with carbamoyl chloride in the presence of K₂CO₃, the reaction was complete in 10-12 h to give octacarbamoylated products **3a,b,e** (Scheme 1, Table 1).



Scheme 1. N,N-Dimethylcarbamoylation of resorcinarenes **1**.

Table 1. Yields, melting points and MALDI data of carbamoylation products **3-5**

No	R	Yield, %	Mp, °C	<i>m/z</i>
3a	CH ₃	42	260-261*	1113.5 [M ⁺]
				1151.1 [M ⁺ +K ⁺]
3b	C ₃ H ₇	68	210-212	1226 [M ⁺]
				1263.9 [M ⁺ +K ⁺]
3e	CH ₂ CH ₂ C ₆ H ₅	40	212-214	1473 [M ⁺]
				1511.8 [M ⁺ +K ⁺]
4a	CH ₃	54	230-232*	1245.8 [M ⁺ +Cs ⁺]
4b	C ₃ H ₇	72	238-240*	1357.7 [M ⁺ +Cs ⁺]
4c	C ₆ H ₁₃	60	198-200	1525 [M ⁺ +Cs ⁺]
4d	C ₉ H ₁₉	57	223-224	1594 [M ⁺ +Cs ⁺]
4e	CH ₂ CH ₂ C ₆ H ₅	70	245-247*	1606 [M ⁺ +Cs ⁺]
5	C ₁₀ H ₇	77(a); 83(b) ^a	290-305*	1561 [M ⁺]

*melting with decomposition; ^athe synthesis was carried out in the presence of K₂CO₃ (a) or Cs₂CO₃(b)

The mass spectra (Table 1) of **3a,b,e** exhibited peaks corresponding to the octacarbamoylated resorcinarenes **3a,b,e** and their complexes with the potassium cation. The ¹H and ¹³C NMR spectra of **3a,b,e** were found to exhibit broadened proton and carbon signals of the resorcinarene core and carbamate groups. This spectral pattern is possible in the case of coexistence of both potassium-coordinated and uncoordinated resorcinarene molecules,

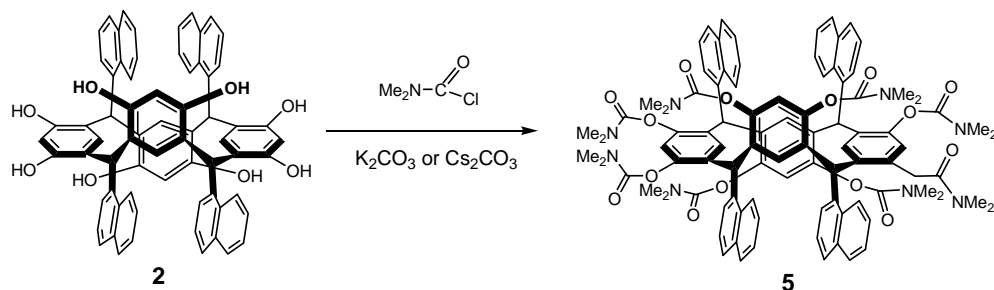
occurring in various conformations owing to retarded *boat-crown-boat* interconversion (Scheme 1). According to elemental analysis and NMR spectroscopy data, the isolated products **3a,b,e** were complexes of the following composition: **3a**•2C₆H₁₄, **3b**•C₆H₁₄, **3e**•C₆H₁₄.

In the presence of Cs₂CO₃, percarbamylation of resorcinarenes **1a-e** with *N,N*-dimethylcarbonyl chloride was complete in 8 h, and the yields of products **4** were higher than those of **3** (Table 1). The difference between the melting/decomposition points of compounds **3a,b,e** and **4a,b,e** is 20–30 °C (Table 1). The mass spectra of **4a-e** exhibited only intense peaks, the spectral parameters corresponding to a 1:1 resorcinarene complex with the cesium cation (Table 1). According to elemental analysis and NMR spectroscopy data, as in the previous case, solvent molecules were incorporated in octacarbamates **4a-e**, apart from CsCl, to form complexes **4a**•CHCl₃•C₆H₁₄, **4b**•2C₆H₁₄, **4c**•C₆H₁₄, **4d**•C₆H₁₄, and **4e**•C₆H₁₄. The ¹H NMR spectra exhibited two singlets for the *ortho*- and *meta*-protons of the benzene rings, one signal for protons of the methylenediphenyl bridge, and four singlets corresponding to the methylamide protons. This spectral pattern is characteristic of *rccc* resorcinarenes existing in the *flattened boat* conformation²². With this spatial arrangement of the octacarbamoylated resorcinarene molecule, the carbamate groups located on the vertically arranged benzene rings are spatially proximate and provide stable ion–dipole interaction with the cesium cation (Scheme 1).

As shown by additional experiments, octacarbamoylated resorcinarenes exhibit selectivity towards cesium cations. The addition of cesium carbonate or hydroxide to a solution of the potassium complex of tetra(phenethyl)resorcinarene **3e** (*m/z* 1511.8 [M⁺+K⁺]) induces displacement of the potassium cation to give complex **4e** with a cesium cation (Scheme 1). The mass spectrum of the isolated product exhibited a peak with a mass number of 1606 corresponding to the complex of octacarbamoylated resorcinarene with cesium. The melting point and spectral data of the isolated product fully coincided with the data obtained previously for complex **4e**.

A similar result was obtained when resorcinarene **1e** was carbamoylated with *N,N*-dimethylcarbonyl chloride in the presence of a mixture of equal amounts of potassium and cesium carbonate (Scheme 1). The reactant ratio was as follows: resorcinarene : carbonyl chloride : K₂CO₃ : Cs₂CO₃ = 1:12:6:6. The physicochemical and spectral characteristics of the isolated product fully corresponded to complex **4e**.

In the carbamoylation of *rctt* tetranaphthylresorcinarene **2**, unlike that of *rccc* resorcinarene **1**, the reaction outcome did not depend substantially on the base chosen (either K₂CO₃ or Cs₂CO₃). In both cases, the reaction ended in the formation of an individual compound: octacarbamoylated derivative **5**, as indicated by elemental analysis and mass spectrometry data (Scheme 2, Table 1).

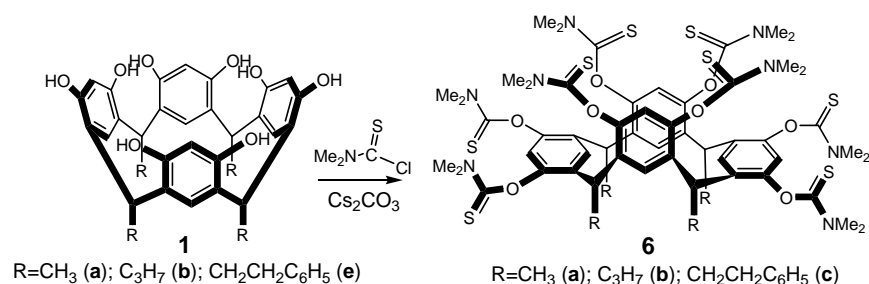


Scheme 2. N,N-Dimethylcarbamoylation of tetranaphthylresorcinarene **2**.

Doubling of the proton and carbon signals of the benzene ring and carbamate groups in the ^1H and ^{13}C NMR spectra of compound **5** and the upfield shift (δ_{H} 5.25 ppm) of the $\text{H}^{3\text{h}}$ protons of the benzene rings in the ^1H NMR spectrum attested to retention of the initial *chair* conformation with $\text{C}_{2\text{h}}$ symmetry.^{23, 24}

The thiocarbamoylation of resorcinarenes **1** and **2** with *N,N*-dimethylthiocarbamoyl chloride was carried out, like carbamoylation, upon refluxing in acetone in the presence of potassium and cesium carbonate. Replacement of the acylating reagent resulted in a sharp increase in the process duration. Completion of the reaction required 48-57 h.

With *rccc* resorcinarenes **1a,b,e** as substrates in the presence of Cs_2CO_3 , the reaction was selective giving only the fully acylated resorcinarenes **6a-c** (Scheme 3). The elemental analysis and mass spectrometry data for **6a-c** correspond to single octathiocarbamoylated derivatives containing no species in the cavity (Table 2). The ^1H and ^{13}C NMR spectra of compounds **6a-c** exhibited proton and carbon signals for the resorcinarene core and the carbamate groups, the integrated intensities of the signals being in line with calculated values, and the IR spectra showed strong $\text{C}=\text{S}$ absorption bands (1170 cm^{-1}).



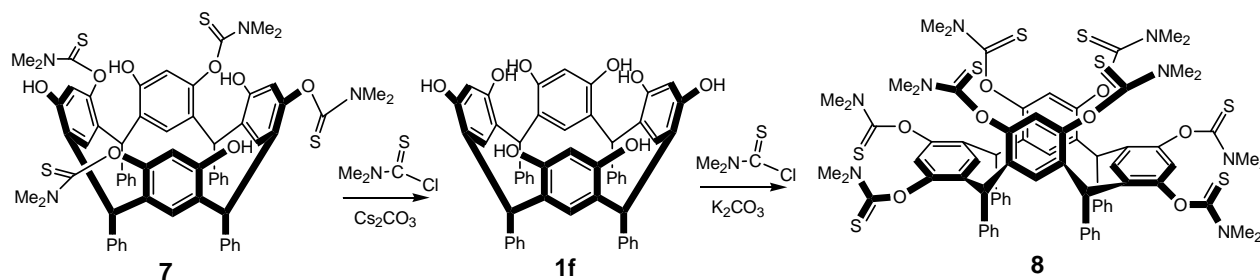
Scheme 3. N,N-Dimethylthiocarbamoylation of resorcinarenes **1a,b,e**.

Table 2. Yields, melting points, and MALDI data of compounds **6-9**

No.	R	Yield, %	M.p., °C	<i>m/z</i>
6a	CH ₃	45	312-314*	1241 [M ⁺]
6b	C ₃ H ₇	31	308-310*	1352 [M ⁺]
6c	CH ₂ CH ₂ C ₆ H ₅	66	196-198	1601 [M ⁺].
7	C ₆ H ₅	58	309-310*	1140 [M ⁺]
8	C ₆ H ₅	54	178-180*	1432 [M ⁺ -4CH ₃]
9	C ₁₀ H ₇	54	248-250*	1690 [M ⁺]

* melting with decomposition

Resorcinarenes **1f**, **2** with aryl substituents R in the lower rim of the macrocycle behaved in a different way. In the case of tetraphenylresorcinarene **1f**, the use of Cs₂CO₃ as the base resulted in tetracarbamoylated product **7** isolated in a 58% yield (Scheme 4). The formation of the tetraacylated derivative was indicated by elemental analysis and mass spectrometry data (Table 2). In the IR spectrum of **7**, C=S absorption band at 1170 cm⁻¹ and OH band at 3228 cm⁻¹ were detected.

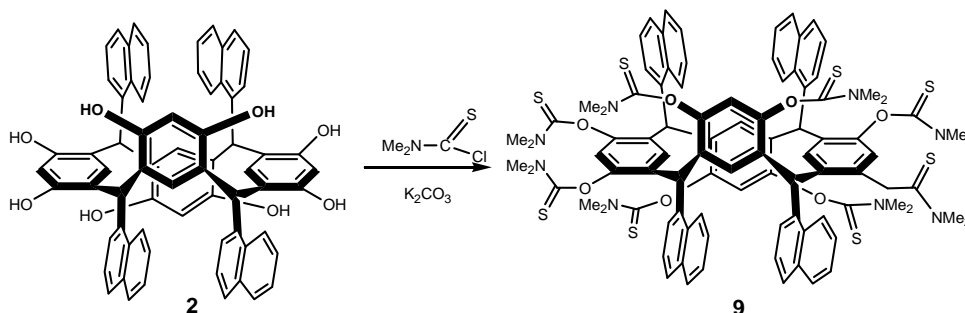
**Scheme 4.** Reaction of tetraphenylresorcinarene **1f** with *N,N*-dimethylthiocarbamoyl chloride.

The NMR data for resorcinarene **7** correspond to a C_{4v}-symmetric molecular structure, which is possible only in the case of a uniform order of immobilization of hydroxyl and thiocarbamoyl groups on all four benzene rings of the macrocycle. The ¹H NMR spectrum of **7** exhibited a singlet for hydrogen atoms of four hydroxyl groups, degenerate proton signals of the benzene rings of the macrocyclic core, and the methylamide proton signals with a 24-proton total integrated intensity. The ¹³C NMR spectrum exhibited a singlet for the thione carbon, two singlets of the carbon atoms of aminomethyl groups, and four singlets for the resorcinol-ring carbon atoms.

By replacing Cs₂CO₃ and K₂CO₃ and increasing the thiocarbamoylation time to 57 h, we obtained perfunctionalized product **8** in the *boat* conformation in 54% yield (Scheme 4). The elemental analysis and mass spectrometry data (Table 2) confirmed the formation of totally acylated resorcinarene **8**. The IR spectrum of **8** exhibited an intense C=S absorption band at 1170

cm^{-1} , and the ^1H NMR spectrum showed no signals for hydroxyl protons. The ^1H and ^{13}C NMR spectra had a more complicated pattern with signal broadening owing to retardation of the *boat-crown-boat* interconversion (Scheme 1) as a result of steric crowding of molecule **8** created by eight bulky functional groups.

The thiocarbamylation of tetranaphthylresorcinarene **2** in the presence of Cs_2CO_3 afforded a mixture of compounds with different degrees of functionalization, which proved to be inseparable. A success was achieved when K_2CO_3 was used, octa(thiocarbamate) **9** was isolated from the reaction mixture in 54% yield (Scheme 5).



Scheme 5. N,N-Dimethylthiocarbamylation of resorcinarene **2**.

The introduction of eight functional groups into *rctt*-resorcinarene **2** with remote benzene rings did not affect the conformation of the macrocyclic core. The elemental analysis, mass spectrometry (Table 2), and IR and NMR spectroscopy fully corresponded to the octa(thiocarbamoyl)resorcinarene **9**.

Thus, we synthesized a new family of oligofunctionalized resorcinarenes with a specific orientation of the carbamate and thiocarbamate groups immobilized on a macrocyclic matrix. It was found that octacarbamoylated *rccc* resorcinarenes in the *boat* conformation exhibit a high capacity for recognition, binding, and recovery of cesium cations. In addition, resorcinarenes containing carbamate and thiocarbamate groups are of interest as potential receptors for organic amines and alcohols.

Experimental Section

General. ^1H and ^{13}C NMR spectra (TMS as an internal standard) were recorded on a Jeol ECX-400 spectrometer operating at 400 MHz for ^1H , 100.5 MHz for ^{13}C . The signals of **3-9** were assigned using H-H homonuclear double resonance (proton spin decoupling). The full assignment of the signals of compounds **3-9** was based on $^1\text{H}/^{13}\text{C}$ 2D correlation. Elemental analysis was performed on Thermo Flash EA112 CHN Elemental analyzer. IR spectra were measured on a NICOLETE 380 Thermo spectrometer in reflection mode in the $4000\text{--}500\text{ cm}^{-1}$ range for samples as Nujol mulls. Resorcinarenes **1** and **2** were synthesized by procedures

reported in ²⁵ and ⁷, respectively.

General procedure of N,N-Dimethylcarbamoylation of resorcinarenes 1 and 2. N,N-Dimethylcarbamoyl chloride (2.65 mmol) was added to a suspension of resorcinarene (0.221 mmol) and metal carbonate (2.65 mmol) in acetone (10 mL). The reaction mixture was stirred at reflux for 40 h (Na₂CO₃), 10-12 h (K₂CO₃), or 8 h (Cs₂CO₃). After that, acetone was fully evaporated, and 10 mL of 5% sulfuric acid was added. The insoluble precipitate was filtered off and washed with 60 mL of water and 20 mL of hexane. The product was kept for 5 h at 80 °C/1 mmHg.

Complex 3a 2C₆H₁₄. Beige powder; yield 0.125 g (42%). decomp. p 260-261 °C. ¹H NMR (CDCl₃): δ 0.87 (t, *J* 7.0 Hz, 12H, CH₃-hexane), 1.29 (m, 16H, CH₂-hexane), 1.46 (d, *J* 6.9 Hz, 12H, CH₃), 2.52 (s, 12H, NCH₃), 2.82 (s, 12H, NCH₃), 3.01 (s, 12H, NCH₃), 3.13 (s, 12H, NCH₃); 4.37 (q, *J* 6.8 Hz, 4H, H¹), 6.01 (s, 2H, H^{3h}), 6.60 (s, 2H, H^{5h}), 6.95 (s, 2H, H^{5v}), 7.32 (s, 2H, H^{3v}). ¹³C NMR (CDCl₃): δ 14.13 (CH₃-hexane), 20.31 (CH₃), 22.72 (CH₂-hexane), 31.60 (CH₂-hexane), 32.26 (C¹), 35.99 (NCH₃), 36.48 (NCH₃), 36.83 (NCH₃), 115.87 (C^{5h}), 116.90 (C^{5v}), 125.26 (C^{3h}), 125.68 (C^{3v}), 131.97 (C^{2v}); 135.68 (C^{2h}), 146.22 (C^{4h}), 148.22 (C^{4v}), 153.89 (C=O), 154.43 (C=O). IR, λ, cm⁻¹: 1701.3 (C=O). MS, *m/z*: 1113.5 [M⁺], 1151.1 [M⁺+K⁺]. Calculated for C₅₆H₇₂N₈O₁₆ KCl 2C₆H₁₄.(%): C 60.05, H 7.41, N 8.24. Found (%): C 60.37, H 7.71, N 8.13.

Complex 3b C₆H₁₄. Beige powder; yield 0.208 g (68%); mp 220-212 °C. ¹H NMR (d₆-DMSO, 25°C): δ 0.83 (t, *J* 7.3 Hz, 12H, CH₂CH₂CH₃. 6H, CH₃-hexane), 1.14 (m, *J* 7.4 Hz, 8H, CH₂CH₂CH₃), 1.28 (m, 8H, CH₂-hexane), 2.03 (m, 8H, CH₂CH₂CH₃), 2.46 (s, 24H, NCH₃), 2.64 (s, 24H, NCH₃), 4.16 (t, *J* 7.4 Hz, 4H, H¹), 6.10 (s, 4H, H³), 7.19 (s, 4H, H⁵). ¹³C NMR (d₆-DMSO, 25 °C): δ 14.14 (CH₃-hexane), 14.39 (CH₃), 20.90 (CH₂), 21.19 (CH₂), 22.70 (CH₂-hexane), 31.63 (CH₂-hexane), 33.09 (C¹), 36.10 (NCH₃), 36.75 (NCH₃), 38.77 (NCH₃), 40.01 (NCH₃), 102.92 (C⁵), 123.78 (C³), 125.48 (C²); 152.11 (C⁴); 154.02 (C=O). MS, *m/z*: 1226 [M⁺], 1263.9 [M⁺+K⁺]. Calculated for C₆₄H₈₈N₈O₁₆ KCl C₆H₁₄ (%): C 60.65, H 7.42, N 8.08. Found (%): C 60.91, H 7.26, N 8.27.

Complex 3e C₆H₁₄. Beige powder; yield 0.143 g (40%). decomp.p 212-214 °C. ¹H NMR (d₆-DMSO, 25 °C): δ 0.85 (t, *J* 6.9 Hz, 12H, CH₃-hexane), 1.27 (m, 16H, CH₂-hexane), 2.23 (m, 8H, CH₂CH₂Ph), 2.55 (br.m, 8H, CH₂CH₂Ph), 2.78 (s, 24H, NCH₃) 2.85 (s, 24H, NCH₃), 4.33 (t, *J* 7.3Hz, 4H, H¹), 7.04 (m, 12H, H³, *o*-Ph), 7.16 (m, 16H, H⁵, *p*, *m*-Ph). ¹³C NMR (d₆-DMSO, 25°C): δ 14.13 (CH₃-hexane), 22.70 (CH₂-hexane), 31.67 (CH₂-hexane), 33.92 (CH₂CH₂Ph), 35.47 (CH₂CH₂Ph), 35.88 (NCH₃), 36.03 (C¹), 36.19 (NCH₃), 116.41 (C⁵), 125.66 (C²), 126.08 (C³), 128.10 (Ph), 128.14 (Ph), 141.39 (C⁴), 153.35 (C=O). IR, λ, cm⁻¹: 1703.6 (C=O). MS, *m/z*: 1473 [M⁺], 1511.8 [M⁺+K⁺]. Calculated for C₈₄H₉₆N₈O₁₆ KCl C₆H₁₄.(%): C 66.14, H 6.78, N 6.86. Found (%): C 66.52, H 6.39, N 6.48.

Complex 4a CHCl₃ C₆H₁₄. Beige powder; yield 0.177 g (54%). decomp.p 230-323 °C. ¹H NMR (CDCl₃): δ 0.86 (t, *J* 7.3 Hz, 12H, CH₃-hexane), 1.26 (m, 16H, CH₂-hexane), 1.46 (d, *J* 6.9 Hz,

12H, CH₃); 2.61 (s, 12H, NCH₃), 2.82 (s, 12H, NCH₃), 3.01 (s, 12H, NCH₃), 3.09 (s, 12H, NCH₃), 4.40 (q, *J* 6.8 Hz, 4H, H¹), 6.01 (s, 2H, H^{3h}), 6.77 (s, 2H, H^{5h}), 7.16 (s, 2H, H^{5v}), 7.37 (s, 2H, H^{3v}). ¹³C NMR (CDCl₃): δ 14.14 (CH₃-hexane), 20.31 (CH₃), 22.72 (CH₂-hexane), 31.61 (CH₂-hexane), 32.26 (C¹); 35.99(NCH₃). 36.48 (NCH₃); 36.83 (NCH₃); 115.87 (C^{5h}), 116.90 (C^{5v}), 125.26 (C^{3h}), 125.68 (C^{3v}), 131.97 (C^{2v}); 135.68 (C^{2h}), 146.22 (C^{4h}), 148.22 (C^{4v}), 153.89 (C=O), 154.43 (C=O). IR, λ, cm⁻¹: 1705.3 (C=O). MS, *m/z*: 1245.8 [M⁺+Cs⁺]. Calculated for C₅₆H₇₂N₈O₁₆ CsCl CHCl₃ C₆H₁₄(%): C 50.88, H 5.90, N 7.53. Found(%): C 50.86, H 6.02, N 7.18.

Complex 4b 2C₆H₁₄. Beige powder; yield 0.249 g (72%); decomp.p 238-240 °C. ¹H NMR (d₆-DMSO, 25 °C): δ 0.83 (t, *J* 7.3 Hz, 12H, CH₂CH₂CH₃, 12H, CH₃-hexane), 1.14 (m, *J* 7.4 Hz, 8H, CH₂CH₂CH₃), 1.28 (m, 16H, CH₂-hexane), 2.03 (m, 8H, CH₂CH₂CH₃), 2.46 (s, 24H, NCH₃), 2.64 (s, 24H, NCH₃), 4.16 (t, *J* 7.4 Hz, 4H, H¹), 6.10 (s, 4H, H³), 7.19 (s, 4H, H⁵). ¹³C NMR (d₆-DMSO, 25 °C): δ 14.16 (CH₃-hexane), 14.39 (CH₃), 20.90 (CH₂), 21.19 (CH₂), 22.70 (CH₂-hexane), 31.64 (CH₂-hexane), 33.09 (C¹), 36.10 (NCH₃), 36.75 (NCH₃), 38.77 (NCH₃), 40.01 (NCH₃), 102.92 (C⁵), 123.78 (C³), 125.48 (C²); 152.11 (C⁴); 154.02 (C=O). MS, *m/z*: 1357.7 [M⁺+Cs⁺]. Calculated for C₆₄H₈₈N₈O₁₆ CsCl 2C₆H₁₄(%): C 58.28, H 7.47, N 7.15. Found(%): C 58.61, H 7.16, N 6.76.

Complex 4c C₆H₁₄. Beige powder; yield 0.219 g (60%). mp. 198-200 °C. ¹H NMR (CDCl₃, 25 °C); δ 0.83 (t, *J* 6.4 Hz, 12H, CH₂(CH₂)₄CH₃, 6H, CH₃-hexane), 1.22 (m, 32H, CH₂(CH₂)₄CH₃, 8H, CH₂-hexane), 1.75 (m, 8H, CH₂(CH₂)₄CH₃), 2.62 (s, 12H, NCH₃), 2.80 (s, 12H, NCH₃), 3.00 (s, 12H, NCH₃), 3.08 (s, 12H, NCH₃), 4.26 (t, *J* 6.8 Hz, 4H, H¹), 6.06 (s, 2H, H^{3h}), 6.69 (s, 2H, H^{5h}), 7.19 (s, 2H, H^{5v}), 7.32 (s, 2H, H^{3v}). ¹³C NMR (CDCl₃, 25°C): δ 14.11 (CH₃, CH₃-hexane), 22.80 (CH₂, CH₂-hexane), 23.05 (CH₂), 28.20 (CH₂), 29.71 (CH₂), 31.64 (CH₂-hexane), 31.84 (CH₂), 33.09 (C¹), 36.47 (NCH₃), 36.60 (NCH₃), 36.74 (NCH₃), 36.78 (NCH₃), 115.88 (C⁵), 117.08 (C⁵), 125.94 (C³), 126.03 (C³), 130.03 (C²), 134.62 (C²), 146.04 (C⁴), 148.75 (C⁴), 153.93 (C=O), 154.28 (C=O). MS, *m/z*: 1525 [M⁺+Cs⁺]. Calculated for C₇₆H₁₁₂N₈O₁₆ CsCl C₆H₁₄(%): C 59.75, H 7.71, N 6.80. Found(%): C 59.84, H 7.37, N 6.68.

Complex 4d C₆H₁₄. Beige powder; yield 0.228 g (57%); mp. 223-224 °C. ¹H NMR (d₆-DMSO, 25 °C), δ, ppm: 0.84 (t, *J* 6.9 Hz, 12H, CH₂(CH₂)₇CH₃, 6H, CH₃-hexane), 1.22 (m, 56H, CH₂(CH₂)₇CH₃, 8H, CH₂-hexane), 2.00 (m, 8H, CH₂(CH₂)₇CH₃), 2.88 (br.s, 48H, NCH₃), 4.17 (t, *J* 7.4 Hz, 2H, H¹), 4.22 (t, *J* 7.6 Hz, 2H, H¹), 6.15 (s, 4H, H³), 7.12 (s, 4H, H⁵). ¹³C NMR (d₆-DMSO, 25 °C): δ 14.14 (CH₃-hexane), 14.40 (CH₃), 21.18 (CH₂), 22.90 (CH₂, CH₂-hexane), 31.64 (CH₂-hexane), 33.09 (C¹), 36.10 (NCH₃), 36.75 (NCH₃), 102.92 (C⁵), 123.72 (C²), 125.48 (C³), 152.11 (C⁴), 154.28 (C=O). MS, *m/z*: 1594 [M⁺+Cs⁺]. Calculated for C₈₈H₁₃₆N₈O₁₆ CsCl C₆H₁₄(%): C 62.15, H 8.32, N 6.17. Found(%): C 62.55, H 8.27, N 6.57.

Complex 4e C₆H₁₄. *Method 1.* The general procedure of carbamoylation of **1e** was used. *Method 2.* Cesium carbonate (0.06 mmol) was added to a solution of complex **3a** (0.06 mmol) in 5 mL of acetone. The suspension was stirred for 3 h at 20 °C. Then acetone was distilled off and the residue was washed with water (10 mL) and hexane (5 mL) and dried for 5 h at 80 °C/1 mmHg. *Method 3.* Cesium hydroxide (0.06 mmol) was added to a solution of complex **3a** (0.06 mmol) in

5 mL of acetone. The resulting suspension was stirred for 3 h at 20 °C. The precipitate formed was filtered off, washed with water (10 mL) and hexane (5 mL), and dried for 5 h at 80 °C/1 mmHg. **Method 4.** Carbamoyl chloride (2.65 mmol) was added to a suspension of resorcinarene **1e** (0.221 mmol), potassium carbonate (1.3 mmol), and cesium carbonate (1.3 mmol) in 10 mL of acetone. The reaction mixture was stirred at reflux for 8 h. After that, acetone was completely distilled off, and 10 mL of 5% sulfuric acid was added to the reaction mixture. The insoluble precipitate was filtered off and washed with 60 mL of water and 20 mL of hexane. The product was dried for 5 h at 80 °C/1 mmHg. Beige powder; yield 0.267 g (70%) (method 1), 0.08 g (79%, method 2), 0.084 g (83%, method 3), 0.317 g (85%, method 4); decomp.p 245-247 °C. ¹H NMR (d₆-DMSO, 25 °C): δ 0.88 (t, *J* 7.3 Hz, 6H, CH₃-hexane), 1.28 (m, 8H, CH₂-hexane), 2.23 (m, 8H, CH₂CH₂Ph), 2.55 (br.m, 8H, CH₂CH₂Ph), 2.78 (s, 24H, NCH₃) 2.85 (s, 24H, NCH₃), 4.33 (t, *J* 7.3Hz, 4H, H¹), 7.04 (m, 10H, H³, *o*-Ph), 7.16 (m, 14H, H⁵, *p*, *m*-Ph). ¹³C NMR (d₆-DMSO, 25 °C): δ 14.12 (CH₃-hexane), 22.68 (CH₂-hexane), 31.65 (CH₂-hexane), 33.92 (CH₂CH₂Ph), 35.47 (CH₂CH₂Ph), 35.88 (NCH₃), 36.03 (C¹), 36.19 (NCH₃), 116.41 (C⁵), 125.66 (C²), 126.08 (C³), 128.10 (Ph), 128.14 (Ph), 141.39 (C⁴), 153.35 (C=O). IR, λ, cm⁻¹: 1718.7 (C=O). MS, *m/z*: 1606 [M⁺+Cs⁺]. Calculated for C₈₄H₉₆N₈O₁₆ CsCl C₆H₁₄(%): C 62.55, H 6.42, N 6.48. Found(%): C 62.84, H 6.27, N 6.68.

Octa(*N,N*-dimethylcarbamoyl)tetranaphthyl-resorcinarene (5). Beige powder; yield 0.265 g (77%, in the presence of K₂CO₃); 0.286 g (83%, in the presence of Cs₂CO₃); mp 290-305 °C. ¹H NMR (CDCl₃, 25 °C): δ 2.24 (s, 12H, NCH₃), 2.35 (s, 12H, NCH₃), 2.68 (s, 12H, NCH₃), 2.92 (s, 12H, NCH₃), 5.25 (s, 2H, H^{3h}), 6.20 (s, 2H, H^{3v}) 6.21 (s, 4H, H¹), 6.50 (d, *J* 6.8 Hz, 4H, H²-Naph), 6.93 (dd, *J* 6.8 Hz, *J* 8.0 Hz, 4H, H⁷-Naph), 6.95 (dd, *J* 7.3 Hz, *J* 6.8 Hz, 4H, H³-Naph), 7.03 (dd, *J* 7.1 Hz, *J* 6.8 Hz, 4H, H⁶-Naph), 7.14 (s, 2H, H^{5h}), 7.17 (s, 2H, H^{5v}), 7.39 (d, *J* 8.0 Hz, 4H, H⁴-Naph), 7.41 (d, *J* 7.8 Hz, 4H, H⁵-Naph) , 7.44 (d, *J* 8.2 Hz, 4H, H⁸-Naph). ¹³C NMR (CDCl₃, 25 °C): δ 34.72 (C¹), 35.25 (NCH₃), 36.13 (NCH₃), 36.14 (NCH₃), 36.21 (NCH₃), 116.70 (C^{5h}), 117.50 (C^{5v}), 124.10 (C-Naph), 124.42 (C-Naph), 124.53 (C-Naph), 124.79 (C-Naph), 126.23 (C-Naph), 126.34 (C-Naph), 127.82 (C-Naph), 128.01 (C-Naph), 128.57 (C^{2v}); 128.55 (C^{2h}); 130.62 (C^{3h}), 130.84 (C^{3v}), 147.74 (C^{4h}); 148.50 (C^{4v}), 152.82 (C=O), 153.77 (C=O). IR, λ, cm⁻¹: 1719.4 (C=O). MS, *m/z*: 1561 [M⁺]. Calculated for C₉₂H₈₈N₈O₁₆(%): C 70.75, H 5.68, N 7.17. Found (%): C 70.77, H 5.65, N 7.15.

General procedure of *N,N*-dimethylthiocarbamoylation of resorcinarenes 1a,b,e,f. *N,N*-Dimethylthiocarbamoyl chloride (3.25 mmol) and cesium carbonate (3.25 mmol) was added to a solution of the specified resorcinarene (0.27 mmol) in 10 mL of acetone. The reaction mixture was stirred for 48 h at 50-55 °C. Then acetone was completely distilled off, and 20 mL of 5% sulfuric acid was added to the residue. The precipitate was filtered off and washed with water (50 mL) and hexane (10 mL). The product was dried for 6 h at 75-80 °C *in vacuo* (1 mmHg).

Octa(*N,N*-dimethylthiocarbamoyl)-tetramethyl-resorcinarene (6a). Beige powder; yield 0.165 g (45%); decomp.p 312-314 °C. ¹H NMR (CDCl₃): δ 1.45 (d, *J* 6.9 Hz, 12H, CH₃), 2.61 (s, 12H, NCH₃), 2.82 (s, 12H, NCH₃), 3.01 (s, 12H, NCH₃), 3.08 (s, 12H, NCH₃), 4.40 (q, *J* 6.8 Hz, 4H, H¹), 6.01 (s, 2H, H^{3h}), 6.77 (s, 2H, H^{5v}), 7.16 (s, 2H, H^{5h}), 7.36 (s, 2H, H^{3v}), ¹³C NMR (d₆-

DMSO): δ 21.13 (CH₃), 31.99 (C¹), 38.73 (NCH₃), 39.04 (NCH₃), 43.15 (NCH₃), 43.54 (NCH₃), 118.65 (C^{5h}), 119.64 (C^{5v}), 125.81 (C^{3h}), 126.20 (C^{3v}), 133.35 (C^{2v}); 136.54 (C^{2h}); 148.47 (C^{4h}); 150.78 (C^{4v}), 185.70 (C=S), 186.48 (C=S). IR, λ , cm⁻¹: 1125.7 (C=S). MS, m/z : 1241 [M⁺]. Calculated for C₅₆H₇₂N₈O₈S₈ CHCl₃(%): C 50.30, H 5.41, N 8.23. Found (%): C 50.68, H 5.46, N 8.38.

Octa(*N,N*-dimethylthiocarbamoyl)-tetrapropyl-resorcinarene (6b). Light yellow powder; yield 0.113 g (31%). decomp.p 308-310 °C. ¹H NMR (d₆-DMSO, 25 °C): δ 0.80 (br.s, 12H, CH₂CH₂CH₃), 1.20 (br.s, 8H, CH₂CH₂CH₃), 1.83 (br.s, 8H, CH₂CH₂CH₃), 2.46 (s, 24H, NCH₃), 2.89 (s, 24H, NCH₃), 4.19 (br.t, 4H, H¹), 6.27 (br.s, 2H, H^{3h}), 6.56 (br.s, 2H, H^{5v}), 6.84 (br.s, 2H, H^{5h}), 7.42 (br.s, 2H, H^{3v}). ¹³C NMR (d₆-DMSO, 25 °C): δ 14.03 (CH₃), 20.77 (CH₂CH₃), 20.93 (CH₂CH₂), 32.20 (C¹), 38.61 (NCH₃), 39.86 (NCH₃), 40.28 (NCH₃), 40.49 (NCH₃), 118.26 (C⁵), 126.49 (C³), 132.10 (C²), 153.79 (C⁴), 185.90 (C=S), 186.16 (C=S). MS, m/z : 1352 [M⁺]. Calculated for C₆₄H₈₈N₈O₈S₈(%): C 56.77, H 6.55, N 8.28. Found (%): C 57.00, H 6.52, N 8.25.

Octa(*N,N*-dimethylthiocarbamoyl)-tetraphenetyl-resorcinarene (6c). Beige powder; yield 0.285 g (66%). mp 196-198 °C. ¹H NMR (CDCl₃, 25 °C): δ 2.31 (m, J 6.9 Hz, J 7.3 Hz, 8H, CH₂CH₂Ph), 2.57 (m, J 7.8 Hz, 4H, CH₂CH₂Ph), 2.75 (m, J 6.4 Hz, 4H, CH₂CH₂Ph), 2.92 (s, 12H, NCH₃), 3.05 (s, 12H, NCH₃), 3.18 (s, 12H, NCH₃), 3.31 (s, 12H, NCH₃), 4.24 (t, J 6.4 Hz, 4H, H¹), 6.61 (s, 2H, H^{3h}), 6.65 (s, 2H, H^{5v}), 6.80 (s, 2H, H^{5h}), 7.06-7.15 (m, 20H, Ph) 7.59 (s, 2H, H^{3v}). ¹³C NMR (CDCl₃, 25 °C): δ 34.19 (CH₂CH₂Ph), 36.27 (CH₂CH₂Ph), 36.54 (C¹), 38.61 (N-CH₃), 39.36 (NCH₃), 42.92 (NCH₃), 43.42 (NCH₃), 119.47 (C^{5h}), 120.63 (C^{5v}), 125.61 (Ph), 127.32 (C^{3h}), 128.68 (Ph), 129.10 (Ph), 129.82 (C^{3v}), 135.22 (C^{2v}), 141.59 (C^{2h}); 148.63 (C^{4h}), 151.77 (C^{4v}), 186.29 (C=S), 186.67 (C=S). IR, λ , cm⁻¹: 1161.9 (C=S), 1100.0 (C=S). MS, m/z : 1601 [M⁺]. Calculated for C₈₄H₉₆N₈O₈S₈(%): C 62.97, H 6.04, N 6.99. Found (%): C 62.61, H 6.02, N 6.63.

Tetrahydroxy-tetra(*N,N*-dimethylthiocarbamoyl)-tetraphenyl-resorcinarene (7). Beige powder; yield 0.1774 g (58%); decomp.p 309-310 °C. ¹H NMR (DMSO, 25 °C): δ 2.93 (s, 12H, NCH₃), 3.24 (s, 12H, NCH₃), 5.58 (s, 4H, H¹), 6.10 (s, 4H, H⁵), 6.10 (s, 4H, H³), 6.69 (m, 8H, Ph), 6.93 (m, 12H, Ph), 8.56 (s, 4H, OH). ¹³C NMR (DMSO, 25 °C): δ 41.90 (C¹), 44.33 (NCH₃), 45.03 (NCH₃), 102.71 (C⁵), 120.89 (C^{Ph}), 125.01 (C^{Ph}), 127.66 (C^{Ph}), 127.84 (C³), 129.09 (C^{Ph}), 146.25 (C²), 153.06 (C⁴), 186.63 (C=S). IR, ν , cm⁻¹: 3402 (OH), 1145 (C=S). MS m/z : 1136 [M⁺]. Calculated for C₆₄H₆₀N₄O₈S₄(%): C, 67.34; H, 5.30; N, 4.91. Found (%): C, 67.43; H, 5.35; N, 4.64.

Octa(*N,N*-dimethylthiocarbamoyl)-tetraphenyl-resorcinarene (8). The synthesis was conducted similarly to the synthesis of **6** by stirring resorcinarene **1f** (1.6448 g, 0.207 mmol), *N,N*-dimethylthiocarbamoyl chloride (3.077 g, 24.9 mmol), and potassium carbonate (2.4651 g, 24.9 mmol) in 10 mL of acetone for 57 h at 50-55 °C. Then acetone was completely distilled off, and 20 mL of 5% sulfuric acid was added to the residue. The product was extracted with CH₂Cl₂ (10 mL), the organic layer was separated, CH₂Cl₂ was distilled off, the residue was dissolved at reflux in a minimum amount of methanol, and the solution was refluxed for 5 min. The methanol-insoluble precipitate was filtered off, washed with hot methanol, and dried for 6 h at

75-80 °C *in vacuo* (1 mmHg). Beige powder; yield 0.3138 g (54%); decomp.p 248-250 °C. ¹H NMR (CDCl₃, 25 °C): δ 2.74 (s, 12H, NCH₃), 2.94 (s, 12H, NCH₃), 3.29 (s, 12H, NCH₃), 3.36 (s, 12H, NCH₃), 5.61 (s, 4H, H¹), 6.31-6.83 (m, 2H, H^{3h}; 2H, H^{5v}; 2H, H^{5h}; 2H, H^{3v}), 6.90-7.10 (br.s, 20H, Ph). ¹³C NMR (CDCl₃, 25 °C): δ 36.72 (NCH₃), 39.05 (NCH₃), 43.28 (NCH₃), 45.09 (NCH₃), 46.82 (C¹), 119.01 (C^{3v}), 126.31 (C^{Ph}), 128.05 (C^{5v,5h}), 129.53 (C^{Ph}), 131.16 (C^{3h}), 140.61 (C^{2v}), 147.56 (C^{2h}), 150.07 (C^{4h}), 154.53 (C^{4h}), 185.46 (C=S). IR, ν, cm⁻¹: 1163 (C=S). MS *m/z*: 1432 [M⁺-4CH₃]. Calculated for C₇₆H₈₀N₈O₈S₈(%): C, 61.26; H, 5.41; N, 7.52. Found (%): C, 61.48; H, 5.35; N, 7.34.

Octa(*N,N*-dimethylthiocarbamoyl)-tetranaphthyl-resorcinarene (9). *N,N*-Dimethylthiocarbamoyl chloride (0.932 g, 7.55 mmol) and potassium carbonate (1.057 g, 7.55 mmol) were added to a solution of tetranaphthylresorcinarene **2** (0.3406 g, 0.343 mmol) in acetone (10 mL). The reaction mixture was stirred for 12 h at room temperature and for 48 h at 50-55°C. Acetone was distilled off, and 20 mL of 5% sulfuric acid was added to the residue. The product was extracted with CH₂Cl₂ (10 mL), and the organic layer was separated and washed with 50 mL of distilled water. Then CHCl₃ was distilled off, 50 mL of hexane was added to the amorphous residue, and the formed powdered precipitate was filtered off and washed with 15 mL of hexane. The product was dried for 6 h at 75-80°C *in vacuo* (1 mmHg). Beige powder; yield 0.3138 g (54%). decomp.p 248-250 °C. ¹H NMR (CDCl₃, 25 °C): δ 2.02 (s, 12H, NCH₃), 2.75 (s, 12H, NCH₃), 3.28 (s, 12H, NCH₃), 3.58 (s, 6H, NCH₃), 3.62 (s, 6H, NCH₃), 6.22 (s, 4H, H¹), 6.32 (s, 2H, H^{3h}), 6.52 (d, *J* 7.3 Hz, 4H, H²-Naph), 6.75 (s, 2H, H^{5v}), 6.79 (s, 2H, H^{5h}), 6.93 (dd, *J* 7.8 Hz; *J* 6.9 Hz, 4H, H⁶-Naph), 6.96 (s, 2H, H^{3v}), 7.01 (dd, *J* 7.8 Hz; *J* 7.3 Hz, 4H, H³-Naph), 7.19 (dd, *J* 7.8 Hz; *J* 6.9 Hz, 4H, H⁷-Naph), 7.44 (d, *J* 8.3 Hz, 4H, H⁴-Naph), 7.61 (d, *J* 8.2 Hz, 4H, H⁸-Naph), 7.8 (d, *J* 8.8 Hz, 4H, H⁵-Naph). ¹³C NMR (CDCl₃, 25 °C): δ 37.48 (NCH₃), 39.56 (C¹), 42.17 (NCH₃), 44.11 (NCH₃), 45.06 (NCH₃), 119.53 (C^{5h}), 120.15 (C^{5v}), 123.7 (C⁵-Naph), 124.02 (C⁷-Naph), 124.99 (C³-Naph), 125.21 (C⁶-Naph), 125.82 (C¹⁰-Naph), 126.6 (C²-Naph), 127.2 (C⁴-Naph), 128.03 (C⁸-Naph), 130.34 (C⁹-Naph), 131.36 (C^{3v}), 132.89 (C^{2v}), 133.37 (C^{3h}), 138.45 (C^{2h}), 149.82 (C^{4h}), 150.48 (C^{4v}), 187.39 (C=S). IR, ν, cm⁻¹: 1160 (C=S), 1125 (C=S). MS *m/z*: 1689 [M⁺]. Calculated for C₉₂H₈₈N₈O₈S₈(%): C, 65.37; H, 5.25; N, 6.63. Found (%): C, 65.54; H, 5.29; N, 6.59.

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