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One-pot Synthesis of Mono-substituted Quaternized *p-tert*-Butylthiacalix[4]arenes

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The possibility of one-pot synthesis of mono-substituted quaternized derivatives of p-tert-butylthiacalix[4]arene was demonstrated for the first time by the reaction of distally di-bromopropyl substituted p-tert-butyl thiacalix[4]arene with several nitrogen-containing nucleophiles. The structure and composition of the reaction products were analyzed by modern physical methods, including two-dimensional NMR spectroscopy and high-resolution mass spectrometry. A detailed analysis of the reaction mixtures made it possible to reveal the dealkylation mechanism, which consists in the nucleophilic attack of the bromide ion on the O-CH_2 carbon atom, followed by the formation of a bromine-containing adduct, detected by mass spectrometry. Dealkylation does not occur when the classical di-bromopropyl substituted p-tert-butyl calix[4]arene is used as a substrate - the bis-quaternized imidazolium calix[4]arene salt is formed in a high yield. Such a difference in the reactivity of two macrocycles is associated with the difference in the sizes of the macrocycles: in the case of di-substituted thiacalixarene, which has a large size, two unsubstituted hydroxyl groups form a hydrogen bond with only one alkoxy group, while in the classical calix[4]arene, two paired hydrogen bonds are formed. As a result, one of the two alkoxy groups of the thiacalixarene is more accessible for nucleophilic attack; formed thereafter thiacalixarene nucleofuge-anion is stabilized by two hydrogen bonds at once.

Keywords: Thiacalix[4]arenes, N-heterocyclic carbene precursors, dealkylation.

Однореакторный синтез моно-замещенных кватернизированных производных *п-трет*-бутилтиакаликс[4]арена

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Впервые путем реакции дистально ди-бромпропил замещенного п-трет-бутилтиакаликс[4]арена с рядом азот-содержащих нуклеофилов продемонстрирована возможность однореакторного синтеза моно-замещенных кватернизированных производных п-трет-бутилтиакаликс[4]арена. Структура и состав продуктов реакций проанализированы комплексом современных физических методов исследования, включающих двумерную ЯМР спектроскопию и масс-спектрометрию высокого разрешения. Детальный анализ реакционных смесей позволил выявить механизм деалкилирования, заключающийся в нуклеофильной атаке бромид-иона на O-CH_2 атом углерода с последующим формированием бром-содержащего аддукта, зафиксированного масс-спектрометрией. При использовании в качестве субстрата классического ди-бромпропил замещенного п-трет-бутилкаликс[4]арена деалкилирования не происходит – формируется бис-кватернизированная имидазолиевая соль с высоким выходом. Подобная разница в реакционной способности двух макроциклов связана с разницей в размерах макроциклов, в результате чего, в случае тиакаликсарена, обладающего большим размером, в ди-замещенных производных две незамещенные гидроксильные группы

образуют водородную связь лишь с одной алкокси-группой, в то время как в классическом каликс[4]арене образуются две парные водородные связи. Вследствие этого, одна из двух алкокси-групп тиакаликсарена оказывается более доступной для нуклеофильной атаки, а сформированный тиакаликсареновый нуклеофуг стабилизируется также сразу двумя водородными связями.

Ключевые слова: Каликс[4]арены, NHC прекурсоры, деалкилирование.

Introduction

In the last decade the attention of many research groups has been focused on the synthesis of N-heterocyclic carbene (NHC) transition metal complexes on a macrocyclic platform.[1,2] The fixation of NHC fragments on the macrocyclic platform allows a significant change in the microenvironment of the metal, opens the opportunity for the varying the number of NHC fragments to give both mono and bis-NHC complexes. The presence of a molecular cavity in the macrocycle allows highly selective metalloenzyme-like transformations due to the possibility of selective inclusion of reagents into the reaction "pocket". Among the variety of macrocycles, a special place is occupied by calix[4]arens and their analogues - thiacalix[4]arens as well as pillar[n]arens.[3-6] These molecules have a number of advantages to build NHC complexes of various structures and compositions on their platform. Modification of both the upper rim of the macrocycles as well as their phenolic hydroxyl groups can be used for introduction of NHC fragments and additional lipophilic fragments to obtain complexes with an amphiphilic architecture. Recently we obtained bis-NHC complexes of palladium(II) on the *p-tert*-butylthiacalix[4]arene platform in the *1,3-alternate* stereoisomeric form.[7] The resulting complexes showed activity both in the Suzuki coupling reaction as well as in the model hydrogenation reaction of *p*-nitrophenol. Continuing research towards the creation of precursors of NHC thiacalix[4]arene complexes, this work presents a new one-pot method for the synthesis of mono-substituted quaternized derivatives of *p-tert*-butylthiacalix[4]arene based on the reaction of available distal-disubstituted O-bromoalkyl derivatives of *p-tert*-butylthiacalix[4]arene with nitrogen containing nucleophiles.

Experimental

The solvents were purified according to known literature methods.[8] All reagents were commercially available from Sigma Aldrich or Alfa-Aesar catalogues. Synthesis of the starting 5,11,17,23-tetra-*tert-butyl*-25,27-dihydroxy-26,28-di-3-bromopropoxy-2,8,14,20-tetrathiacalix[4]arene (**1**) and 5,11,17,23-tetra-*tert-butyl*-25,27-dihydroxy-26,28-di-3-bromopropoxy-2,8,14,20 tetracalix[4]arene (**5**) was carried out according to the literature methods.[9,10]

The purity of the substances was controlled by thin layer chromatography, which was carried out using silica gel coated plates manufactured by Merck (HX68558954) with Vilber Lourmat VL-6.LC UV lamp control at 254 nm. NMR spectra were recorded at 25 °C on a Bruker Nanobay spectrometer (400 MHz for ^{1}H and 101 MHz for ^{13}C). Chemical shifts were corrected relative to the signals of residual protons of deuterated solvents $CDCl_3$ (for ^{1}H 7.26 ppm; for ^{13}C 77.36 ppm). IR spectra of the compounds were recorded on an IR Fourier spectrometer Bruker Vector-22 in the range of 400-4000 cm^{-1}. Samples were prepared using suspending in mineral oil or as thin films, obtained from chloroform solutions dried on the surface of the KBr tablet. High-resolution mass spectra with electrospray ionization (HRESI MS) were obtained on an Agilent iFunnel 6550 Q-TOF LC/MS. Carrier gas - nitrogen, temperature 300 °C, carrier flow rate 12 L/min, nebulizer pressure 275 kPa, funnel voltage 3500 V, capillary voltage 500 V, total ion current recording mode, 100-3000 *m/z* mass range, scanning speed 7 spectra/s. The melting points of the substances were determined on an OptiMelt MPA100 automatic heating table (Oxford Instruments, USA). Elemental analysis was performed on a EuroVector EA 3000 CHN analyzer.

General procedure for the quaternization reaction: 0.18 mmol of dibromopropyloxy derivative of *p-tert*-butyl(thia)calix[4]arene (**1** or **5**) and 1.8 mmol of tertiary amine (pyridine, 1-methylimidazole, N-methylmorpholine) were placed into the glass autoclave 'GlassChem' (CEM ® corporation) and dissolved in 3 mL of dry acetonitrile. The reactions were carried out in an inert nitrogen atmosphere. The reaction mixture was heated to 130 °C for 16-60 hours. The progress of the reaction was monitored by TLC (petroleum ether: ethyl acetate 1:4, the resulting salts have $R_f = 0$). To isolate the target products, the solvent was evaporated to give precipitate, which was dried in *vacuo* for 8 hours. The product was additionally purified by column chromatography (eluent methanol:methylene chloride 1:7).

5,11,17,23-Tetra-tert-butyl-25,26,27-trihydroxy-28-(3-(3-N-methylimidazolium)propoxy)-2,8,14,20-tetrathiacalix[4]arene bromide, ***2***. Yield: 0.14 g (88%). HRESI MS: *m/z* $[M-Br]^+$ calcd. $C_{47}H_{59}N_2O_4S_4^+$ 843.3353, found: 843.3345. T_m (decomp) = 245 °C. IR (KBr) ν_{max} cm^{-1}: 1261 (CAr -O), 1571 (C = N), 2869 (CH_2), 2961 (CH_3). ^{1}H NMR ($CDCl_3$, 363 K) δ_H ppm: 0.67 (9H, s, $C(CH_3)_3$), 1.21 (9H, s, $C(CH_3)_3$), 1.29 (18H, s, $C(CH_3)_3$), 2.48 (2H, p, CH_2, J = 5.7 Hz), 4.11 (3H, s, CH_3), 4.57 (2H, t, CH_2O, J = 5.7 Hz), 4.94 (2H, br.t, CH_2N, J = 5.7 Hz), 6.83 (2H, s, ArH), 7.20 (2H, br.s, ImdH), 7.55 (2H, s, ArH), 7.58 (2H, d, ArH, J = 2.5 Hz), 7.63 (2H, d, ArH, J = 2.5 Hz), 10.43 (1H, s, HImd). ^{13}C NMR ($CDCl_3$) δ ppm: 30.56, 31.05, 31.68, 37.09, 47.42, 68.75, 122.12, 122.84, 122.95, 123.45, 123.65, 129.83, 131.32, 134.43, 134.98, 136.26, 139.58, 147.19, 156.69, 158.84.

5,11,17,23-Tetra-tert-butyl-25,26,27-trihydroxy-28-(3-(1-pyridinium)propoxy)-2,8,14,20-tetrathiacalix[4]arene bromide, ***3***. Yield: 0.13 g (76%). HRESI MS: *m/z* $[M-Br]^+$ calcd. for $C_{48}H_{58}NO_4S_4^+$ 840.3244, found 840.3247. T_m (decomp) = 200 °C. IR (KBr) ν_{max} cm^{-1}: 1251 (CAr -O), 1633 (C = N), 2869 (CH_2), 2962 (CH_3). ^{1}H NMR ($CDCl_3$, 363 K) δ_H ppm: 0.64 (9H, s, C $(CH_3)_3$), 1.21 (9H, s, C $(CH_3)_3$), 1.30 (9H, s, C $(CH_3)_3$), 2.75 (2H, p, CH_2, J = 5.1 Hz), 4.55 (2H, t, CH_2O, J = 5.1 Hz), 5.29 (2H, br.t, CH_2N, J = 5.1 Hz), 6.80 (2H, s, HAr), 7.56 (2H, s, HAr), 7.60 (2H, br.d, HAr), 7.64 (2H, br.d, HAr), 8.07-8.13 (2H, m, PyH), 8.17-8.23 (1H, m, PyH), 10.00 (2H, m, PyH). ^{13}C NMR ($CDCl_3$) δ ppm: 30.57, 31.70, 34.16, 59.37, 67.96, 122.07, 123.36, 123.58, 128.61, 129.97, 131.37, 134.50, 135.03, 136.31, 141.26, 144.36, 146.94, 147.50, 156.40, 158.66.

5,11,17,23-Tetra-tert-butyl-25,26,27-trihydroxy-28-(3-(3-N-methylmorpholinium)propoxy)-2,8,14,20-tetrathiacalix[4]arene bromide, ***4***. Yield: 0.14 g (84%). HRESI MS *m/z* $[M-Br]^+$ calcd. for $C_{48}H_{64}NO_5S_4^+$ 862.3663, found: 862.3662; T_m (decomp) = 200 °C. IR (KBr) ν_{max} cm^{-1}: 1260 (CAr -O), 1570 (C-N), 2869 (CH_2), 2962 (CH_3). ^{1}H NMR ($CDCl_3$, 363 K) δ_H ppm: 0.46 (9H, s, $C(CH_3)_3$), 1.19 (9H, s, $C(CH_3)_3$), 1.31 (18H, s, $C(CH_3)_3$), 2.44-2.65 (2H, m, CH_2), 3.50 (3H, s, CH_3), 3.67-3.73 (4H, m, CH_2N), 3.97-4.12 (4H, m, CH_2O), 4.34 (4H, br.t, CH_2O), 4.75 (4H, br.t, CH_2N^+), 6.54 (2H, s,

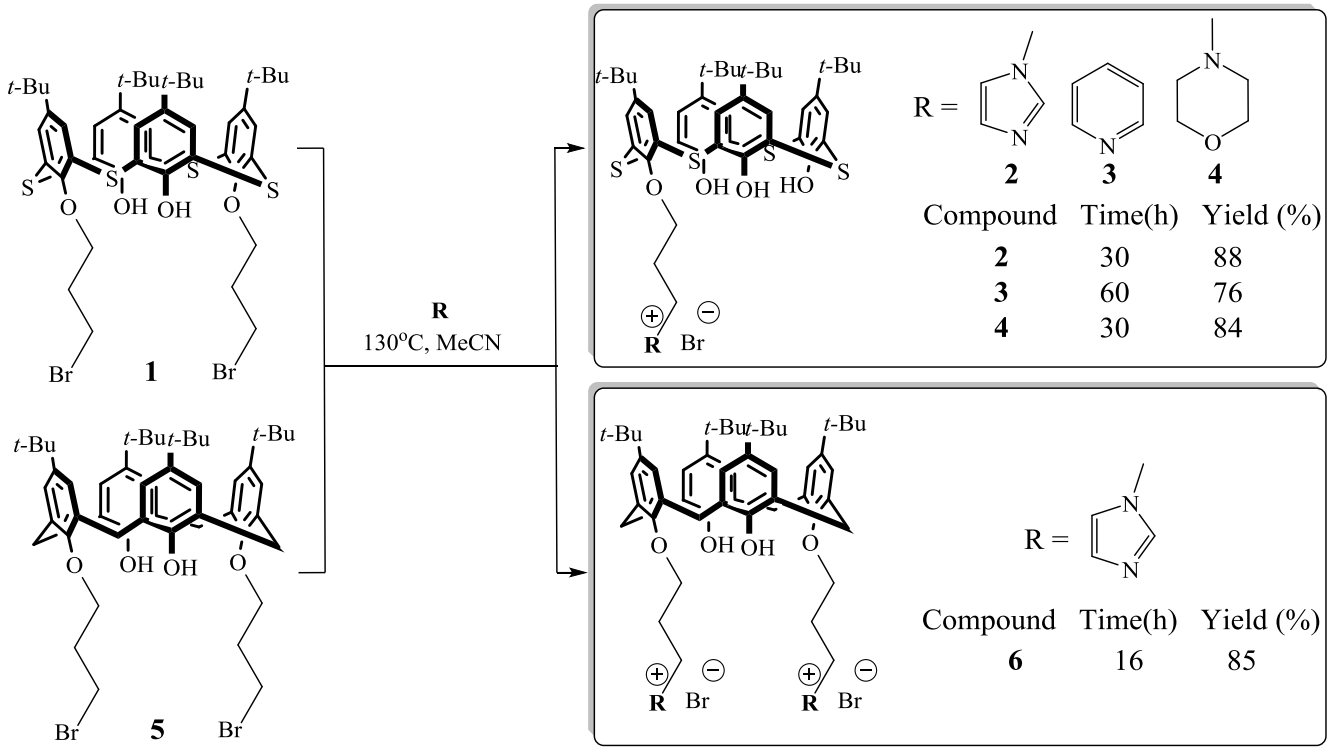
ArH), 7.49 (2H, s, ArH), 7.58 (4H, s, ArH). ^{13}C NMR ($CDCl_3$) δ ppm: 23.89, 29.85, 30.32, 34.19, 47.27, 60.48, 61.07, 121.91, 123.82, 124.14, 129.28, 130.68, 134.14, 134.81, 136.29, 141.42, 147.52, 155.53, 157.79.

5,11,17,23-Tetra-tert-butyl-25,27-dihydroxy-26,28-bis(3-(3-N-methylimidazolium)propoxy)-2,8,14,20-calix[4]arene dibromide, **6**. Yield: 0.16 g (85%). HRESI MS *m/z* $[M-2Br]^{2+}$ calcd. for $C_{58}H_{78}N_4O_4^{2+}$ 447.3006, found 447.3008. T_m (decomp) = 235 °C. IR (KBr) ν_{max} cm^{-1}: 1240 (CAr -O), 1573 (C = N), 2869 (CH_2), 2962 (CH_3). ^{1}H NMR ($CDCl_3$, 363 K) δ_H ppm: 0.97 (18H, s, C $(CH_3)_3$), 1.28 (18H, s, C $(CH_3)_3$), 2.68 (4H, p, CH_2, J = 7.0 Hz), 3.36 (4H, d, CH_2, J = 13.0 Hz), 4.05-4.15 (14H, m, CH_2, OCH_2, CH_3), 4.84 (4H, t, CH_2N, J = 6.9 Hz), 6.83 (4H, s, ArH), 7.07 (4H, s, ArH), 7.47 (2H, s, OH), 7.53 (2H, br.s, ImdH), 7.85 (2H, br.s, ImdH), 10.26 (2H, s, ImdH). ^{13}C NMR ($CDCl_3$) δ ppm: 31.07, 31.78, 31.96, 34.03, 34.13, 37.11, 46.79, 72.08, 122.67, 124.09, 125.56, 125.98, 127.82, 132.33, 137.85, 142.74, 147.84, 149.32, 149.99.

Results and Discussion

In order to obtain new precursors for bis-NHC complexes on the platform of *p-tert*-butylthiacalix[4]arene and classical *p-tert*-butylcalix[4]arene, the reaction of dibromopropyl-substituted calix[4]arenes with N-methylimidazole was carried out. For this purpose, the initial distal-disubstituted *O*-bromopropyl-containing macrocycles **1** and **5** were obtained according to the literature methods[9,10] by the Mitsunobu reaction of the initial thia- or classic calix[4]arenes with bromopropanol. The obtained macrocycles **1** and **5** were used in Menshutkin's reaction with 1-methylimidazole according to Scheme 1. The reactions were carried out in acetonitrile in a 'GlassChem' autoclave (CEM® corporation) at 130 °C with the thin layer chromatography control of the reaction.

As a result of the quaternization reaction between dibromo-substituted thiacalix[4]arene **1** with 1-methylimidazole, after 30 hours of stirring monosubstituted adduct **2** was isolated in high yield (88%) instead of the expected di-substituted product. The structure of the adduct **2** was proved by NMR, IR spectroscopy, high-resolution electrospray mass spectrometry (HRESI MS). Dealkylation during quaternization is unambiguously evidenced by the data of both one-dimensional and two-dimensional ^{1}H-^{1}H NOESY NMR spectroscopy and HRESI MS experiments. Thus, the ^{1}H NMR spectrum of the macrocycle **2** (Figure 1) exhibits a typical signal pattern of mono-substituted thiacalixarenes (two singlets and two doublets of signals for aromatic protons 7, 9, 8, and 8 ', as well as three signals for protons of *tert-butyl* groups 10, 11 and 12 as singlets with a ratio of 1:2:1). In the 2D (^{1}H-^{1}H) NOESY NMR spectrum of compound **2**, there are the cross-peaks between the signals of N-CH-N protons of the imidazolium fragment 1 (δ = 10.41 ppm) and methylene protons of the propyl linker 4-6 (δ = 4.91, 2.47 and 4.57 ppm) and also between the signals of the N-CH_3 protons of fragment 2 (δ = 4.06 ppm) and the nearby imidazolium protons 3, 3 'and 1 (δ = 7.21 and 10.41 ppm). The presence of cross peaks between the protons of neighboring *tert-butyl* groups (δ = 1.20 and 0.63; 1.29 and 0.65 ppm) indicates the "*cone*" stereoisomeric form of macrocycle **2**.



Scheme 1. Synthesis of imidazolium salts based on *p-tert*-butyl(thia)calix[4]arene.

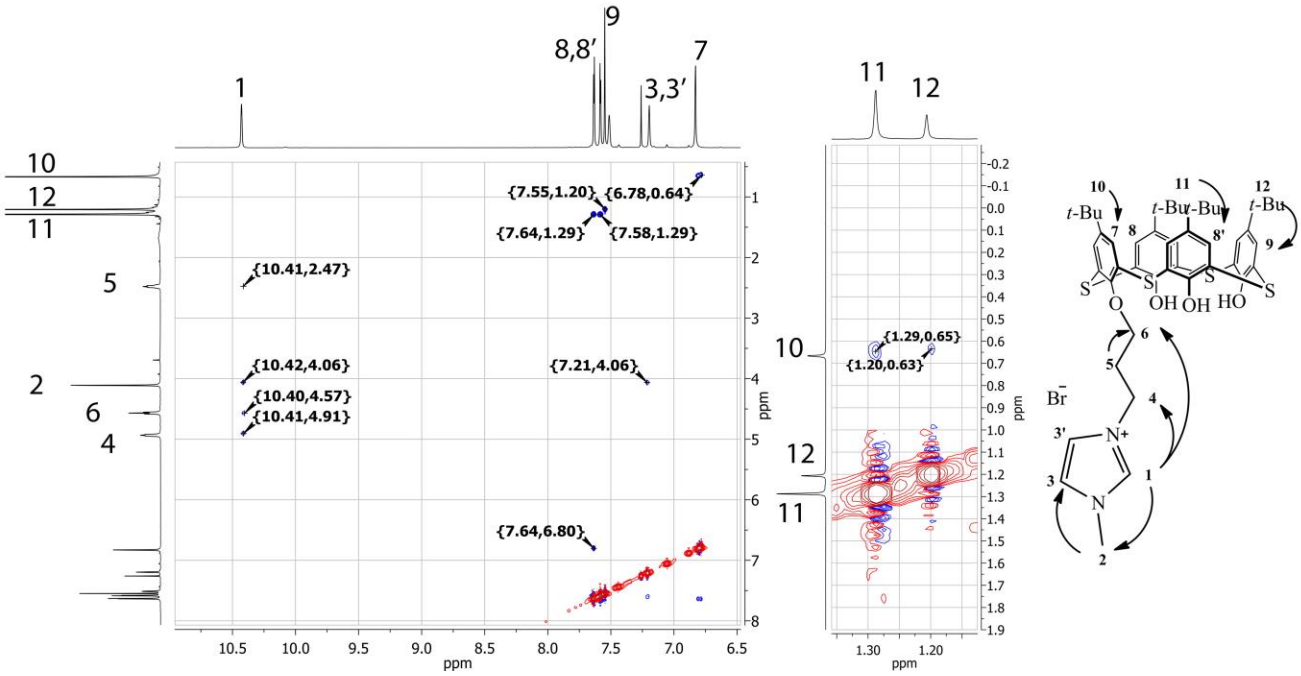


Figure 1. Fragments of the 2D (^{1}H-^{1}H) NOESY NMR spectrum of compound **2** ($CDCl_3$, 25 °C).

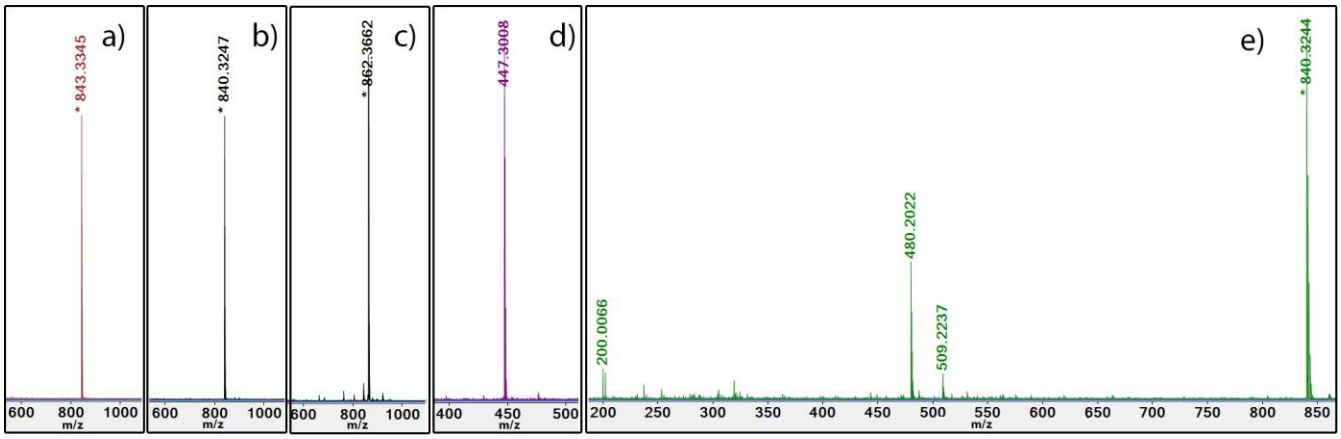
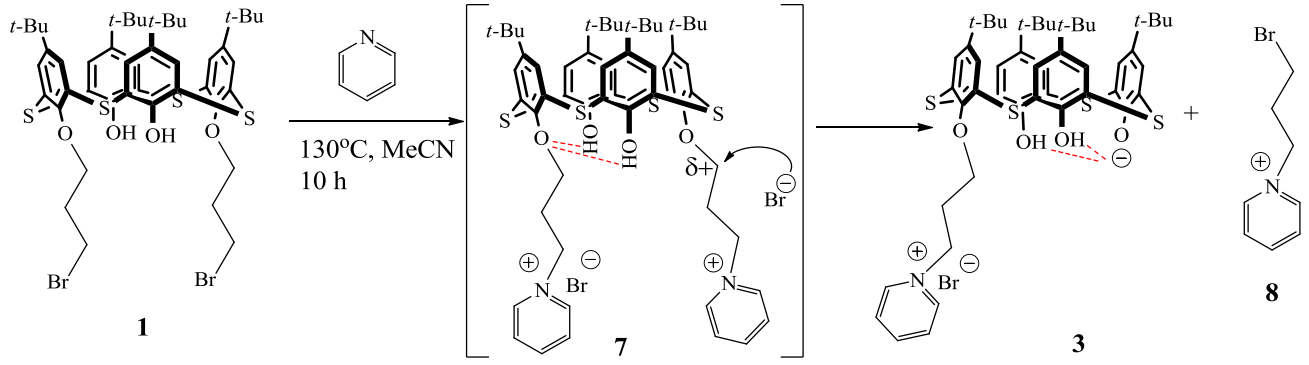


Figure 2. Fragments of HRESI mass spectra of compounds **2** (a), **3** (b), **4** (c), **6** (d), as well as reaction mixture **1** with pyridine after 10 hours of heating (e).

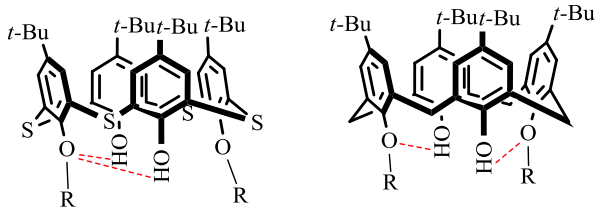
The reaction with other nucleophiles (pyridine and N-methylmorpholine) proceeds in a similar way - as the only product, monosubstituted salts **3** and **4** were also isolated in high yields. The structure of all compounds was also confirmed by HRESI MS. The mass spectrum (Figure 2) of compound **2** showed a peak $[M\text{-}Br]^+$ with *m/z* = 843.3345 (calculated for $C_{47}H_{59}N_2O_4S_4^+$ 843.3353), similar peaks of the molecular ion $[M\text{-}Br]^+$ were recorded for compound **3**: *m/z* = 840.3247 (calculated for $C_{48}H_{58}NO_4S_4^+$ 840.3244) and **4**: *m/z* = 862.3662 (calculated for $C_{48}H_{64}NO_5S_4^+$ 862.3663).

To reveal the mechanism of the observed dealkylation, the reaction mixture of **1** with pyridine was analyzed by HRESI MS after 10 hours of reflux. According to the data obtained (Figure 2e), the mixture contains a peak $[M\text{-}2Br]^{2+}$ of di-quaternized product **7** with *m/z* = 480.2022 (calculated for $C_{56}H_{68}N_2O_4S_4^{2+}$ 480.2026) (Scheme 2) and mono-quaternized product **3**. In addition to these signals, the spectrum contains $[M\text{-}Br]^+$ signal of the adduct **8** with *m/z* = 200.0066 (calculated for $C_8H_{11}BrN^+$ 200.0070).

The above HRESI MS data indicate that the mechanism of the dealkylation reaction involves a nucleophilic attack of the alpha-carbon atom of the ether group with a bromide ion, which leads to the cleavage of the carbon-oxygen bond, the formation of 1-(3-bromopropyl)pyridinium bromide **8** and the elimination of the nucleofuge - the anion of monosubstituted calixarene **3**, which is then protonated during the treating of the reaction mixture with water (Scheme 2). It is known that the high nucleophilicity of the bromide ion



Scheme 2.



Scheme 3.

allows cleavage of aryloxy-alkyl bonds with the release of the corresponding phenols in almost quantitative yield.[11] Dialkyl-substituted derivatives of *p-tert*-butylthiacalix[4]arene also undergo selective mono-dealkylation both in the presence of an excess of tetraalkylammonium halides[12] and in the presence of other nucleophiles (amines,[13] sodium azide[14]). It is noteworthy that no dealkylation occurs during the quaternization of calix[4]arene **5** with 1-methylimidazole: after 16 hours of reflux, the distally disubstituted product **6** was isolated in a high yield (85%). The ^{1}H NMR spectrum of compound **6** exhibits a classic pattern of signals characteristic of distally disubstituted calix[4]arenes: two singlets at δ = 1.28 and 0.97 ppm, corresponding to the protons of *tert-butyl* groups, two singlets at δ = 6.83 and 7.07 ppm, corresponding to the protons of the aromatic rings of calix[4]arene, as well as one signal of OH - protons at 7.47 ppm. According to HRESI MS data, the composition of compound **6** is fully consistent with the proposed structure - the peak $[M-2Br]^{2+}$ with *m/z* = 447.3008 (calculated for $C_{58}H_{78}N_4O_4^{2+}$ 447.3006) is observed. This result clearly demonstrates the unique hydrolytic instability inherent in thiacalix[4]arenes compared to classical calix[4]arenes, as discussed earlier.[15] Due to the fact that the length of the bond between the carbons of the benzene ring and sulfur in the thiacalix[4]arene is 1.77 Å, while in the classical calix[4]arene the bond between the carbons of the benzene ring and the methylene fragment is 1.54 Å, the cavity of the thiacalix[4]arene is about 15% larger than that of calix[4]arene.[16] Such a cavity size difference leads to the different structures of distally disubstituted products as in the crystalline phase[17] and in solution.[18] For thiacalix[4]arene, the most characteristic conformation is the "*distorted cone*", in which two hydrogens of phenolic hydroxyl groups form a hydrogen bond with only one alkyloxy fragment, while all oxygen atoms of classical calixarene are involved in the hydrogen bond (Scheme 3) and the most preferred conformation is the "*flattened cone*".

As a result of the distortion of the thiacalix[4]arene structure, one of the two substituents becomes more sterically available for nucleophilic attack. Stabilization of the nucleofuge is also important: in the case of thiacalix[4]arene, two hydrogen atoms of phenolic hydroxyl groups take part in the stabilization by a hydrogen bond (Scheme 3).

Conclusions

Convenient synthesis of mono-substituted quaternized salts by reaction of distal-dibromopropyl substituted derivatives of thiacalix[4]arene with nitrogen-containing nucleophiles was demonstrated for the first time. The reaction mechanism including nucleophilic attack of bromide ion to alpha-carbon atom of the ether group was evaluated by the HRESI MS method. It has been demonstrated that dealkylation occurs only with the participation of thiacalix[4]arene, while the expected di-substituted product is formed with the use of classical calix[4]arene.

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