Methodology of C(sp^2)—H functionalization in mono- and diazine *N*-oxides in the synthesis of heterocyclic *meso*-substituted calixarenes*

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Earlier unknown *meso*-substituted azinylcalixarenes were obtained by a direct cross-coupling of 2-lithium-25,26,27,28-tetramethoxycalix[4]arenes with mono- and diazine *N*-oxides without transition metal catalysis.

Key words: C—H functionalization, S_NH reactions, calixarenes, azine *N*-oxides.

The C—H-functionalization methodology[1—3] in azaheterocycles, being an atom-economical[4,5] alternative to the transition metal-catalyzed cross-coupling[6—8] of organometal agents with halo-containing substrates, is an efficient synthetic tool for the incorporation of function-bearing fragments into the structure of azines and their activated forms, *i.e.*, *N*-oxides. One of the versions of the C(sp^2)—H bond functionalization methodology, requiring no transition metal (generally, palladium) catalysis and the presence of a halogen or other easily leaving groups in the structure of the starting substrates, are the reactions of nucleophilic hydrogen substitution S_NH (see Refs 9—11).

Earlier, we reported a successful application of the S_NH-methodology in the chemistry of macrocyclic compounds, calix[4]arenes. A direct coupling of tetramethoxycalix[4]arene *meso*-lithium derivatives with 1,2,4-triazines resulted in the obtaining of earlier unknown derivatives hatarylated at the bridging fragment, which are stable σ^H-adducts, and the corresponding aromatic S_NH-products in good yields.[12]

The present work is devoted to the studies of the synthetic potential of the charge-activated forms of mono- and diazines, *N*-oxides, in the synthesis of *meso*-substituted heteroarylcalixarenes. An increased interest in such compounds is caused by the fact that the derivatives based on calixarenes[13—15] possessing various useful properties can be used as ion-carriers through the semipermeable membranes[16—17], receptors for the selective extraction of metal ions,[18] materials for nonlinear optics,[19] catalysts,[20] physiologically active compounds,[21] *etc.*

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Results and Discussion

It is known that 25,26,27,28-tetramethoxycalix[4]arenes treated with Bu^nLi in the presence of the chelating agent TMEDA can be deprotonated at the bridging CH_2 group with the formation of stable lithium derivatives.[22—24] In the present work, we studied the reactivity of 2-lithium-25,26,27,28-tetramethoxycalix[4]arene (**1**), serving as a nucleophile, in the reactions with the structurally various mono- and diazine *N*-oxides **2a—f** having mono-, bi-, or tricyclic systems (Scheme 1). In accordance with the generally accepted notions about nucleophilic heteroaromatic substitution of hydrogen, this transformation is a two-step process, which follows the "addition—elimination" scheme (S_NH(AE)). In the first step, the nucleophilic reagent **1** adds to the C=N bond of the azine *N*-oxide **2a—f** with the formation of low stable intermediate compounds, σ^H-adducts **3a—f**. In the second step, the oxygen-containing fragment eliminates together with the proton at the sp^3-hybridized carbon atom of the azaheterocycle with the formation of aromatized S_NH-products **4a—f**. The use of AcCl in the aromatization step allowed us to obtain the reaction products in 44—74% yields. This can be explained by the fact that the acetate anion is a better nucleofuge as compared to the hydroxide anion.

The synthesized azinylcalixarenes **4a—f** were isolated by column chromatography on SiO_2 and characterized by elemental analysis, mass spectrometry, ^1H and ^{13}C NMR and IR spectroscopy.

Methoxy-substituted calix[4]arenes are known to possess a high conformational lability in many organic solvents.[23] This circumstance imposes certain limitations on

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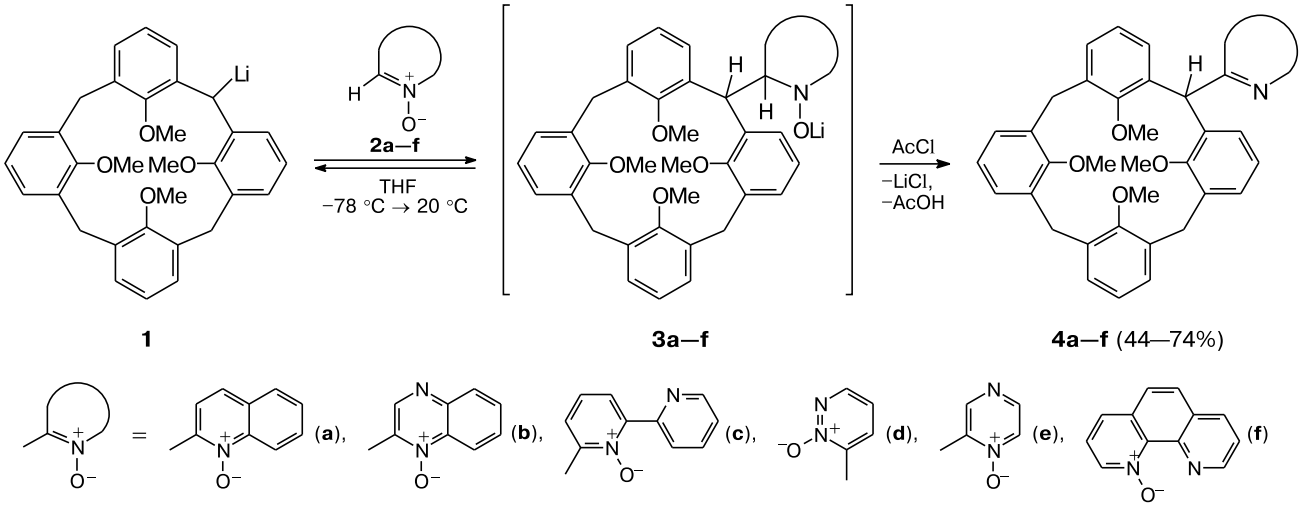
Scheme 1



the structural studies of these compounds by NMR spectroscopy. One of the possible solutions to this problem is the immobilization of the macrocycles in a certain conformation through the formation of a complex of calixarenes methoxy-substituted at the lower rim with Na^+ ion. This procedure consists in the addition of a solution of NaI in CD_3CN to a solution of calixarene in $CDCl_3$. Despite this approach showed a positive result in the case of triazinyl-substituted methoxycalixarenes,[12] in the case of mono- and diazinyl-containing calixarenes, a certain conformation was successfully immobilized only for quinoxaline **4b** and 2,2′-bipyridyl **4c** derivatives. The ^{1}H NMR spectra of these compounds exhibit a single set of resonance signals. In the case of quinoline **4a**, pyridazine **4d**, pyrazine **4e**, and 1,10-phenanthroline **4f** derivatives, several sets of broad signals are observed in the ^{1}H NMR spectra, which is consistent with the existence of these macrocycles in several conformational states.

In the ^{1}H NMR spectra, the signals for the CH_2 and the OMe protons are found in the region δ 2.90—4.55. The resonance multiplet signals for the aromatic protons are observed in the region δ 6.19—9.25. The *meso* protons at the sp^3-hybridized carbon atoms resonate in the region δ 5.78—6.78. In the ^{13}C NMR spectra, the signals for the CH_2 carbons are found in the region δ 27.4—31.7, for the sp^3-hybridized carbons in the region δ 38.1—47.6, for the carbons of the OMe groups in the region δ 57.8—65.4, for the carbons of the (hetero)aromatic fragments in the region δ 121.3—171.1. The mass spectra exhibit peaks of the molecular ions $[M]^+$.

In conclusion, we showed that the methodology of nucleophilic C—H functionalization of azine *N*-oxides can be successfully used for the synthesis of earlier unknown *meso*-substituted heteroarylcalixarenes.

Experimental

^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra were recorded on a Bruker Avance II spectrometer in a mixture of CD_3CN—$CDCl_3$ (2 : 8), using Me_4Si as an internal standard. IR spectra were recorded on a Perkin Elmer Spectrum One B FTIR spectrometer with a diffusion reflection appliance (DRA) for neat samples. Electron impact (EI) mass spectra were obtained on a Shimadzu GCMS-QP2010 Ultra EI instrument. Elemental analysis was performed on a Perkin Elmer 2400-II CHNS/O analyzer. Melting points were measured on a Boetius apparatus. The R_f values were measured on Alugram Sil G/UV-254 precoated plates (Macherey—Nagel). Merck silica gel 60 (0.063—0.200 mm) was used for column chromatography.

Solvents were purified and dried according to the standard procedures. The reagent Bu^nLi (1.6 *M* in hexane) was purchased from Alfa Aesar. Other reagents and solvents are of domestic production.

25,26,27,28-Tetramethoxycalix[4]arene,[25] *N*-oxides of quinoline **2a**, quinoxaline **2b**, pyridazine **2d**, pyrazine **2e** (see Ref. 26), 2,2′-bipyridyl **2c** (see Ref. 27), and 1,10-phenanthroline **2f** (see Ref. 28) were synthesized by the oxidation of the corresponding azines according to the procedures described in the literature.

Synthesis of *meso*-substituted calixarenes 4a—f (general procedure). A 1.6 *M* solution of Bu^nLi in hexane (1.44 mL, 2.3 mmol) was added to a vigorously stirred solution of TMEDA (0.43 mL, 2.9 mmol) in anhydrous THF (4 mL) cooled to −78 °C. After 40 min of stirring, a solution of 25,26,27,28-tetramethoxycalix[4]arene (480.5 mg, 1.0 mmol) in anhydrous THF (5 mL) was added, the reaction mixture was warmed up to room temperatures and stirred for another 2 h. The solution of formed 2-lithium-25,26,27,28-tetramethoxycalix[4]arene (**1**) was cooled to −78 °C, and a solution of the corresponding azine *N*-oxide **2a—f** (2.0 mmol) in anhydrous THF (6 mL) was added. The reaction mixture was stirred at room temperature for 1 h, followed by the addition of AcCl (0.14 mL, 2.0 mmol). The mixture was concentrated at reduced pressure. The residue was subjected to column chromatography on SiO_2, the eluate was concentrated dry *in vacuo*.

2-(Quinolin-2-yl)-25,26,27,28-tetramethoxycalix[4]arene (4a). The yield was 0.437 g (72%), m.p. 270—272 °C. Eluent hexane—EtOAc (9 : 1), R_f 0.30. ^{1}H NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 3.15—3.32 (m, 4 H, 1 H ArC$\underline{H}_2$Ar + 3 H OMe); 3.36—3.45 (m, 2 H, ArC$\underline{H}_2$Ar); 3.65—3.97 (m, 9 H, OMe); 4.16—4.19 (m, 1 H, ArC$\underline{H}_2$Ar); 4.39—4.44 (m, 2 H, ArC$\underline{H}_2$Ar); 6.19—6.35 (m, 5 H, (Het)Ar); 6.39, 6.78 (both s, 1 H, C(sp^3)—H); 6.52—6.72 (m, 3 H, (Het)Ar); 6.90—7.13 (m, 3 H, (Het)Ar); 7.27—7.54 (m, 3 H, (Het)Ar); 7.61—7.75 (m, 1 H, (Het)Ar); 8.07—8.27 (m, 2 H, (Het)Ar); 8.77—8.88 (m, 1 H, (Het)Ar). ^{13}C NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 27.4 (CH_2); 28.9—29.3 (CH_2); 30.3 (CH_2); 38.1, 38.6 (C(sp^3)); 57.8 (OMe); 58.3 (OMe); 59.5 (OMe); 59.6 (OMe); 121.3; 121.4; 121.5; 121.6; 123.7; 123.9; 125.4; 125.5; 126.8; 127.2; 127.9; 128.1; 129.2; 129.3; 131.1; 131.5; 132.2; 132.7; 132.9; 135.9; 138.0; 147.4; 147.6; 147.7; 147.9; 149.2; 156.1; 156.5; 156.7; 157.3. IR (DRA), ν/cm^{-1}:732; 766; 791; 827; 1010; 1086; 1167; 1204; 1248; 1283; 1389; 1423; 1462; 1505; 1569; 1592; 2820; 2856; 2931; 3016; 3064. MS (ESI), *m/z* (I_{rel} (%)): 607 [M]$^+$ (100). Found (%): C, 80.88; H, 5.94; N, 1.99. $C_{41}H_{37}NO_4$. Calculated (%): C, 81.03; H, 6.14; N, 2.30.

2-(Quinoxalin-2-yl)-25,26,27,28-tetramethoxycalix[4]arene (4b). The yield was 0.450 g (74%), m.p. 210—212 °C. Eluent hexane—EtOAc (8 : 2), R_f 0.30. ^{1}H NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 3.43—3.60 (m, 3 H, ArC$\underline{H}_2$Ar); 3.72—4.02 (m, 2 H, ArC$\underline{H}_2$Ar); 4.19—4.43 (m, 13 H, 1 H ArC$\underline{H}_2$Ar + 12 H OMe); 6.61 (s, 1 H, C(sp^3)—H); 6.84—7.02 (m, 4 H, (Het)Ar); 7.08—7.30 (m, 6 H, (Het)Ar); 7.41—7.61 (m, 2 H, (Het)Ar); 7.71—7.87 (m, 2 H, (Het)Ar); 8.00—8.15 (m, 2 H, (Het)Ar); 8.91 (s, 1 H, HetAr). ^{13}C NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 29.5 (CH_2); 29.7 (CH_2); 30.4 (CH_2); 41.2 (C(sp^3)); 64.4 (OMe); 64.8 (OMe); 65.1 (OMe); 65.4 (OMe); 125.6; 126.0; 126.1; 126.3; 129.1; 129.3; 129.4; 129.6; 129.7; 130.2; 134.6; 135.0; 135.1; 135.2; 136.3; 141.1; 141.7; 145.4; 145.5; 145.6; 153.0; 153.3; 153.5; 153.6; 156.3; 156.4; 156.5. IR (DRA), ν/cm^{-1}: 729; 764; 799; 833; 924; 953; 1009; 1088; 1110; 1126; 1171; 1206; 1249; 1287; 1349; 1426; 1464; 1488; 1560; 1588; 2818; 2868; 2929; 2985; 3016; 3069. MS (ESI), *m/z* (I_{rel} (%)): 608 [M]$^+$ (100). Found (%): C, 79.24; H, 6.20; N, 4.49. $C_{40}H_{36}N_2O_4$. Calculated (%): C, 78.92; H, 5.96; N, 4.60.

2-(2,2´-Bipyridin-6-yl)-25,26,27,28-tetramethoxycalix[4]arene (4c). The yield was 0.279 g (44%), m.p. 218—220 °C. Eluent hexane—EtOAc (8 : 2), R_f 0.20. ^{1}H NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 3.21—3.98 (m, 6 H, 3 H ArC$\underline{H}_2$Ar + 3 H OMe); 4.08—4.31 (m, 10 H, 1 H ArC$\underline{H}_2$Ar + 9 H OMe); 4.32—4.47 (m, 2 H, ArC$\underline{H}_2$Ar); 6.33 (s, 1 H, C(sp^3)—H); 6.67—7.27 (m, 13 H, (Het)Ar); 7.28—7.34 (m, 1 H, (Het)Ar); 7.77—7.87 (m, 1 H, HetAr); 8.23—8.31 (m, 1 H, HetAr); 8.32—8.41 (m, 1 H, HetAr); 8.53—8.65 (m, 2 H, HetAr). ^{13}C NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 29.9 (CH_2); 30.8 (CH_2); 31.7 (CH_2); 43.3 (C(sp^3)); 61.6 (OMe); 61.7 (OMe); 62.0 (OMe); 62.1 (OMe); 121.5; 122.5; 122.7; 123.7; 125.0; 133.8; 135.1; 135.5; 136.4; 137.0; 149.3; 149.4; 153.5; 156.2; 156.5; 158.1; 164.7. IR (DRA), ν/cm^{-1}: 729; 743; 761; 795; 1012; 1087; 1163; 1204; 1248; 1397; 1426; 1461; 1552; 1567; 1583; 1599; 2820; 2871; 2925; 3018; 3061. MS (EI), *m/z* (I_{rel} (%)): 634 [M]$^+$ (100). Found (%): C, 79.12; H, 6.34; N, 4.16. $C_{42}H_{38}N_2O_4$. Calculated (%): C, 79.47; H, 6.03; N, 4.41.

2-(Pyridazin-3-yl)-25,26,27,28-tetramethoxycalix[4]arene (4d). The yield was 0.355 g (54%), m.p. 186—188 °C. Eluent hexane—EtOAc (4 : 6), R_f 0.20. ^{1}H NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 3.10—3.52 (m, 5 H, 2 H ArC$\underline{H}_2$Ar + 3 H OMe); 3.54—4.01 (m, 11 H, 2 H ArC$\underline{H}_2$Ar + 9 H OMe); 4.23—4.46 (m, 2 H, ArC$\underline{H}_2$Ar); 5.76, 6.18 (both s, 1 H, C(sp^3)—H); 6.20—6.45 (m, 2 H, (Het)Ar); 6.48—7.32 (m, 10 H, (Het)Ar); 7.36—7.42 (m, 1 H, HetAr); 9.05—9.12 (m, 1 H, HetAr); 9.13—9.29 (m, 1 H, (Het)Ar). ^{13}C NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 30.3—30.8 (CH_2); 40.4—40.7 (C(sp^3)); 60.4 (OMe); 61.3 (OMe); 61.8—62.2 (OMe); 121.7; 122.5; 122.8; 126.8; 128.1; 128.3; 129.2; 130.2; 132.2; 133.3; 133.9; 134.5; 134.7; 135.3; 135.7; 137.1; 143.1; 151.1; 153.8; 157.9; 171.1. IR (DRA), ν/cm^{-1}: 744; 766; 806; 838; 930; 1001; 1089; 1111; 1118; 1174; 1206; 1255; 1289; 1352; 1421; 1463; 1477; 1561; 1590; 2820; 2866; 2944; 2988; 3020; 3071. MS (ESI), *m/z* (I_{rel} (%)): 608 [M]$^+$ (86). Found (%): C, 79.98; H, 5.77; N, 4.29. $C_{44}H_{38}N_2O_4$. Calculated (%): C, 80.22; H, 5.81; N, 4.25.

2-(Pyrazin-2-yl)-25,26,27,28-tetramethoxycalix[4]arene (4e). The yield was 0.234 g (42%), m.p. 180—182 °C. Eluent hexane—EtOAc (8 : 2), R_f 0.20. ^{1}H NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 2.90—3.89 (m, 16 H, 4 H ArC$\underline{H}_2$Ar + 12 H OMe); 4.15—4.53 (m, 2 H, ArC$\underline{H}_2$Ar); 5.87, 6.25 (both s, 1 H, C(sp^3)—H); 6.29—7.04 (m, 12 H, (Het)Ar); 8.39 (m, 1 H, HetAr); 8.45—8.74 (m, 2 H, HetAr). ^{13}C NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 30.2—31.0 (CH_2); 43.3—43.8 (C(sp^3)); 61.0—62.1 (OMe); 122.4; 122.5; 122.6; 127.2; 128.1; 128.7; 129.1; 133.7; 134.9; 135.3; 135.7; 142.2; 144.2; 145.6; 157.4; 157.8; 157.9; 160.0. IR (DRA), ν/cm^{-1}: 767; 779; 831; 1012; 1081; 1155; 1202; 1252; 1285; 1391; 1425; 1462; 1504; 1577; 1593; 2820; 2866; 2932; 3017; 3066. MS (ESI), *m/z* (I_{rel} (%)): 558 [M]$^+$ (100). Found (%): C, 77.12; H, 5.92; N, 4.97. $C_{36}H_{34}N_2O_4$. Calculated (%): C, 77.40; H, 6.13; N, 5.01.

2-(1,10-Phenanthrolin-2-yl)-25,26,27,28-tetramethoxycalix[4]arene (4f). The yield was 0.447 g (68%), m.p. 188—190 °C. Eluent hexane—EtOAc (6 : 4), R_f 0.30. ^{1}H NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 3.09—3.38 (m, 3 H, ArC$\underline{H}_2$Ar); 3.39—3.55 (m, 1 H, ArC$\underline{H}_2$Ar); 3.56—3.27 (m, 12 H, OMe); 4.33—4.55 (m, 2 H, ArC$\underline{H}_2$Ar); 6.34, 6.72 (both s, 1 H, C(sp^3)—H); 6.36—6.84 (m, 9 H, (Het)Ar); 6.88—7.15 (m, 2 H, (Het)Ar); 7.22—7.38 (m, 1 H, HetAr); 7.55—7.67 (m, 1 H, HetAr); 7.68—7.92 (m, 3 H, (Het)Ar); 8.11—8.19 (m, 1 H, (Het)Ar); 8.19—8.32 (m, 1 H, (Het)Ar); 9.10—9.25 (m, 1 H, (Het)Ar). ^{13}C NMR ($CDCl_3$—CD_3CN (8 : 2)), δ: 30.4—31.0 (CH_2); 31.7 (CH_2); 47.3—47.6 (C(sp^3)); 61.0 (OMe); 61.5—61.9 (OMe); 62.0—62.4 (OMe); 122.4; 122.9; 123.9; 126.1; 126.7; 127.2; 127.8; 128.2; 129.1; 130.4; 132.4; 133.9; 135.5; 136.1; 136.3; 137.8; 146.3; 150.2; 158.2; 163.2. IR (DRA), ν/cm^{-1}: 723; 764; 811; 833; 859; 1006; 1085; 1167; 1204; 1246; 1297; 1392; 1420; 1462; 1490; 1505; 1554; 1587; 1619; 2819; 2866; 2928; 2981; 3016; 3059. MS (ESI), *m/z* (I_{rel} (%)): 658 [M]$^+$ (55). Found (%): C, 79.92; H, 6.16; N, 4.13. $C_{44}H_{38}N_2O_4$. Calculated (%): C, 80.22; H, 5.81; N, 4.25.

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