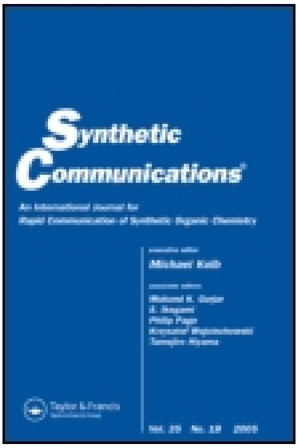

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Novel Triphenylamine Appended 1,3-Alternate-Calix[4]arenes: Synthesis and Characterization

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NOVEL TRIPHENYLAMINE APPENDED 1,3-*ALTERNATE*-CALIX[4]ARENES: SYNTHESIS AND CHARACTERIZATION

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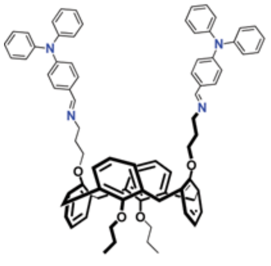
GRAPHICAL ABSTRACT



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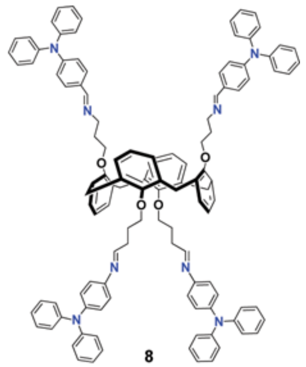

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Abstract *In the present study, two novel calix[4]arene receptors containing triphenylamine units in 1,3-*alternate *conformation have been synthesized and characterized in detail. First, the 25,27-dipropoxy-26,28-bis[(3-aminopropyl)oxy]-calix[4]arene* **4** *and 25,26,27,28-tetra[(3-aminopropyl)oxy]-calix[4]arene* **7** *were prepared by using convenient reagents. Then, these amino derivatives of calix[4]arene were converted to Schiff base derivatives appended to triphenylamine of calix[4]arene (***5** *and* **8***) using 4-formyltriphenylamine via condensation. The 1,3-*alternate *conformation of the synthesized calix[4]arenes was determined by ^{1}H and ^{13}C NMR analyses. Also, their structures have been characterized by using ^{1}H and ^{13}C NMR, infrared, and elemental analyses.*

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Keywords Calix[4]arene; characterization; Schiff base; synthesis; triphenylamine

INTRODUCTION

Calixarenes (Fig. 1) is one of the most important macrocyclic host molecules in supramolecular chemistry,[1,2] together with crown ethers[3] and cyclodextrins.[4]

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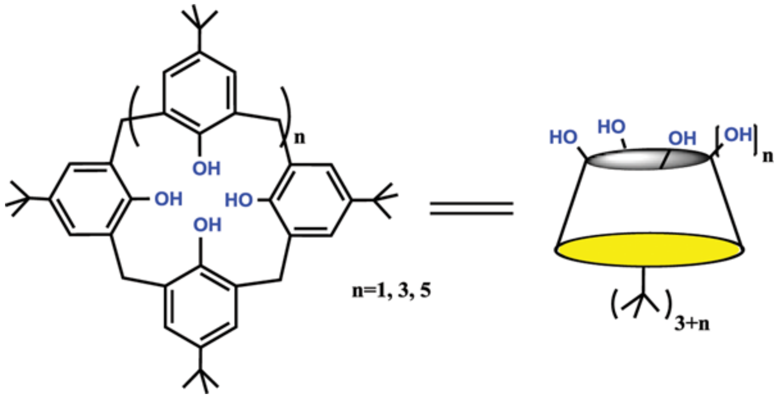
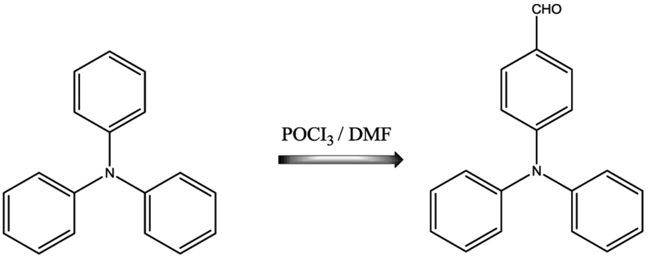


Figure 1. The general molecular structures of calix[*n*]arenes. (Figure is provided in color online.)

They are prepared by the base-catalyzed reaction of formaldehyde with phenol derivatives. Calixarenes have generated considerable interest as useful building blocks for the synthesis of hosts for cations, anions, and neutral molecules. During the past two decades, they have attracted much attention as receptors in supramolecular chemistry. The increasing interest in these compounds is stimulated by the simple large-scale synthesis of calixarenes, and the different ways in which they can be selectively functionalized at the narrow (phenolic groups) or at the wide rim (aromatic nuclei). One of the most important features of these compounds is their diversity. Calixarenes find applications as selective binders and carriers, as analytical sensors, as catalysts, and as model structures for biomimetic studies.[5–8]

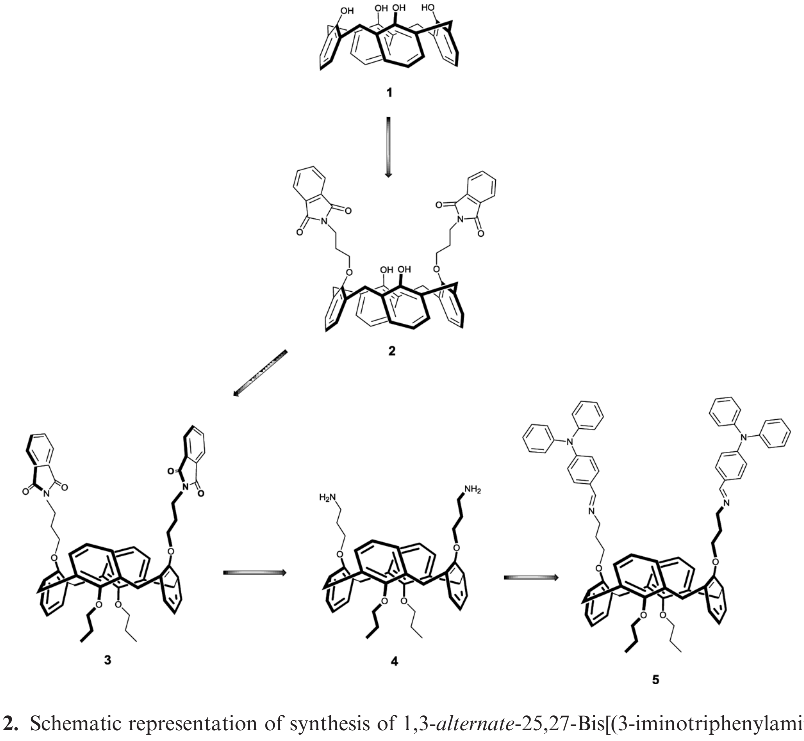
Considerable studies have been made on organic light-emitting diodes (OLEDs) for potential applications in next-generation full-color displays as a promising alternative to the conventional liquid-crystal displays.[9–12] Among the various kinds of OLED materials, triarylamine derivatives have been widely investigated for almost two decades because these compounds have showed excellent thermal and electrochemical stability, electron-donating ability, and optoelectronic properties,[13–17] and they are standard hole-transport materials.[18–22] Considerable effort in synthetic chemistry, in particular by Shirota and coworkers, has led to the development of many classes of triphenylamine-based compounds as hole-transporting or electroluminescent materials.[23,24] To our knowledge, most triphenylamine derivatives are connected by the N–C single bond and obtained either through palladium-catalyzed cross-coupling reactions requiring expensive and extremely air-sensitive palladium catalysts or through copper-catalyzed Ullmann condensations usually involving high temperatures and prolonged reaction times.[25–28] Few papers about triphenylamine derivatives connected by the C–C double bond have been published.[29,30] Recently, the synthesis of different compounds containing triphenylamine and their luminescent characteristics have been reported, whereas the calixarene derivatives bearing triphenylamine have not been synthesized until now. We think that they could be interesting as organic light-emitting diodes (OLEDs). Therefore we report the synthesis and characterization of the new triphenylamine-bearing calix[4]arene derivatives in the present study.



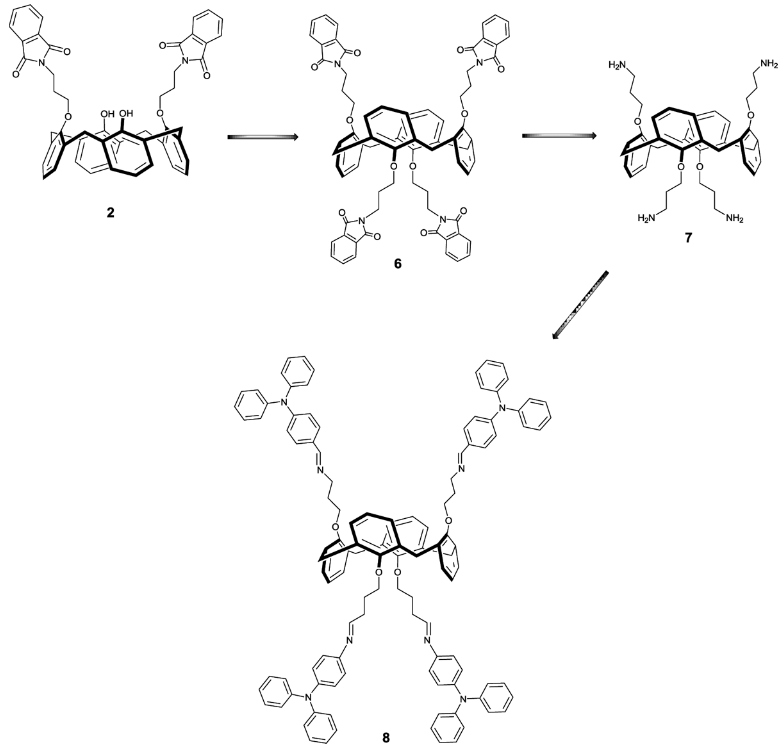
Scheme 1. Synthesis of 4-formyltriphenylamine.

RESULTS AND DISCUSSION

The main focuses of this work are the synthesis and characterization of new 1,3-*alternate*-calix[4]arene receptors bearing triphenylamine. To achieve the desired goal, first we have synthesized 4-formyltriphenylamine using phosphorus oxychloride and dimethylformamide (DMF) according to the literature procedure[31] (Scheme 1). Two novel calix[4]arene Schiff base derivatives containing triphenylamine groups were synthesized by using 25,27-dipropoxy-26,28-bis[(3-aminopropyl)oxy]calix[4]arene **4** or 25,26,27,28-tetra[(3-aminopropyl)oxy]-calix[4]arene **7** and 4-formyltriphenylamine as the precursor. The synthetic routes for these Schiff bases are shown in Scheme 2 and 3. Calix[4]arene **1**, 25,27-bis[(3-phthalimidopropyl)oxy]-calix[4]arene **2**, 25,27-



Scheme 2. Schematic representation of synthesis of 1,3-*alternate*-25,27-Bis[(3-iminotriphenylaminepropyl)oxy]-26,28-dipropoxy calix[4]arene **5**.



Scheme 3. Schematic representation of the synthesis of 1,3-*alternate*-25,26,27,28-tetra[(3-iminotriphenylaminepropyl)oxy]-calix[4]arene **8**.

dipropoxy-26,28-bis[(3-phthalimidopropyl)oxy]-calix[4]arene **3**, and 25,27-dipropoxy-26,28-bis[(3-aminopropyl)oxy]-calix[4]arene **4** were synthesized according to the reported literature procedures.[6,32] Compound **4** was refluxed with 4-formyltriphenylamine and a catalytic amount of triethylamine in $CHCl_3$–methanol to give the corresponding Schiff base derivative of calix[4]arene **5** in 80% yield. Scheme 3 shows the pathway for the synthesis of 1,3-*alternate*-25,26,27,28-tetra[(3-iminotriphenylaminepropyl)oxy]-calix[4]arene **8**. To obtain 25,26,27,28-tetra[(3-phthalimidopropyl)oxy]-calix[4]arene **6** in a 65% yield, 25,27-bis[(3-phthalimidopropyl)oxy]-calix[4]arene **2** was reacted with N-(3-bromopropyl)phthalimide in the presence of Cs_2CO_3. Subsequently, the phthalimido groups in compound **6** were removed with hydrazine hydrate in ethanol to give 25,26,27,28-tetra[(3-aminopropyl)oxy]-calix[4]arene **7** in a 90% yield. Then, 25,26,27,28-tetra[(3-iminotriphenylaminepropyl)oxy]-calix[4]arene **8** was obtained by reacting compound **7** with 4-formyltriphenylamine and a catalytic amount of triethylamine in $CHCl_3$–methanol.

The structures of the synthesized compounds were characterized by a combination of Fourier-transform infrared (FTIR), ^1H NMR, ^{13}C NMR, and elemental analyses. The formation of **5** and **8** was confirmed by the appearance of imine protons (CH=N) at 8.18 and 8.13 ppm in ^1H NMR spectra, respectively (see the Supporting Information, available online). The synthesis of Schiff base derivatives of calix[4]arene (**5** and **8**) was also confirmed by the appearance of the characteristic HC=N bands at about 1641 and

1640 cm^{-1} and by the disappearance of the NH$_2$ band at 3368 and 3369 cm^{-1} in their FTIR spectra, respectively. The ^{13}C NMR spectra of compound **5** and **8** show the appearance of carbon signals at 160.26 and 160.44 ppm belonging to C=N groups of Schiff base, respectively (see the Supporting Information, available online).

The conformational characteristics of the synthesized calix[4]arenes were conveniently estimated by the splitting pattern of the ArCH$_2$Ar methylene protons in the ^{1}H and ^{13}C NMR spectra. ^{1}H and ^{13}C NMR spectra showed that **5** and **8** have a 1,3-*alternate* conformation. The 1,3-*alternate* conformation of **5** and **8** was confirmed by the carbon absorption of the bridging methylene groups (ArCH$_2$Ar) of the calix[4]-arene present at 36.68 and 37.31 ppm in the ^{13}C NMR and the singlet around 3.66 and 3.71 ppm in the ^{1}H NMR for the corresponding methylene hydrogens,[33] respectively.

To summarize, we have designed and synthesized two novel calix[4]arene receptors, **5** and **8**, which have two and four triphenylamine units at a narrow rim of the calix[4]arene moiety. The availability as OLED material of the prepared new receptors will be investigated soon.

EXPERIMENTAL

Melting points were determined on an Electrothermal 9100 apparatus in a sealed capillary and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded at room temperature on a Varian 400-MHz spectrometer in CDCl$_3$. FTIR spectra were obtained on a Perkin-Elmer Spectrum 100 FTIR spectrometer. Elemental analyses were performed using a Leco CHNS-932 analyzer. Analytical thin-layer chromatography (TLC) was performed using Merck prepared plates (silica gel 60 F254 on aluminum). All reactions, unless otherwise noted, were conducted under a nitrogen atmosphere. All starting materials and reagents used were of standard analytical grade from Fluka, Merck, and Aldrich and used without further purification.

1,3-*Alternate*-25,27-bis[(3-iminotriphenylaminepropyl)oxy]-26,28-dipropoxy-calix[4]arene 5

1,3-*Alternate*-25,27-Bis[(3-aminopropyl)oxy]-26,28-dipropoxy-calix[4]arene **4** (1.0 g, 1.61 mmol) in CHCl$_3$ (30 mL) was added to a stirred solution of 4-formyltriphenylamine (0.92 g, 3.38 mmol) and triethylamine (1 mL, excess) in methanol (20 mL), and the mixture was stirred for 24 h under nitrogen. After evaporation of the solvent, the solid residue was washed with cool methanol twice to remove unreacted 4-formyltriphenylamine and dried in vacuum. Yield 80%; mp 209–213 °C; FTIR; 1641 cm^{-1} (HC=N); ^{1}H NMR (400 MHz, CDCl$_3$): δ (ppm) 0.88 (t, 6H, $J = 7.4$ Hz, CH$_2$CH_3), 1.57 (h, 4H, $J = 7.4$ Hz, OCH$_2$CH_2CH$_3$), 1.95 (p, 4H, $J = 7.0$ Hz, OCH$_2$CH_2CH$_2$N), 3.49 (t, 4H, $J = 7.2$ Hz, CH$_2$CH_2N), 3.59 (2 × t, 8H, $J = 6.8$ Hz and $J = 7.0$ Hz, OCH_2CH$_2$CH$_2$N and OCH_2CH$_2$CH$_3$), 3.66 (s, 8H, ArCH_2Ar), 6.68–6.72 (m, 4H, ArH), 7.01 (d, 4H, $J = 7.4$ Hz, ArH), 7.04–7.07 (m, 12H, ArH), 7.10–7.13 (m, 8H, ArH), 7.59 (d, 4H, $J = 8.0$ Hz), 8.18 (s, 2H, CHN). ^{13}C NMR (100 MHz, CDCl$_3$): δ (ppm) 160.26, 156.52, 149.95, 147.20, 133.72, 129.93, 129.77, 129.72, 129.36, 129.00, 125.08, 123.58, 122.16, 121.66, 121.61, 73.31, 69.79, 58.38, 36.68, 31.74, 23.36, 10.40. Calculated for C$_{78}$H$_{76}$N$_4$O$_4$: C, 82.65%; H, 6.76%; N, 4.94%. Found: C, 82.72%; H, 6.81%; N, 5.01%.

1,3-*Alternate*-25,26,27,28-tetra[(3-phthalimidopropyl)oxy]-calix[4]arene 6

A solution of 25,27-bis[(3-phthalimidopropyl)oxy]calix[4]arene 2 (1.0 g, 1.25 mmol), Cs_2CO_3 (1.6 g, 4.9 mmol), and N-(3-bromopropyl)phthalimide (2.0 g, 7.5 mmol) in dry MeCN (100 mL) was refluxed for 3 days. Then, the solvent was removed under reduced pressure. Subsequently, the residue was dissolved in CH_2Cl_2 (70 mL) and washed with 1 N NH_4Cl (3 × 50 mL) and water (2 × 50 mL). The organic phase was separated, dried with $MgSO_4$, and evaporated to afford a solid, which was crystallized from MeOH to give **6** as a pure white powder. Yield 65%; mp 255–257 °C; FTIR; 1694 cm^{-1} (C=O); ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.79–7.65 (m, 16H, PhH), 6.98 (d, 8H, $J = 7.4$ Hz, ArH), 6.82 (t, 4H, $J = 7.4$ Hz, ArH), 3.72 (s, 8H, $ArCH_2Ar$), 3.52 (t, 8H, $J = 7.2$ Hz, NCH_2), 3.45 (t, 8H, $J = 7.4$ Hz, OCH_2), 1.59 (p, 8H, $J = 7.2$ Hz, $OCH_2CH_2CH_2N$); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 168.3, 156.6, 134.0, 133.9, 132.4, 129.6, 123.2, 122.9, 68.4, 38.0, 35.4, 29.0. Calculated for $C_{72}H_{60}N_4O_{12}$: C, 73.71%; H, 5.15%; N, 4.78%. Found: C, 73.76%; H, 5.21%; N, 4.83%.

1,3-*Alternate*-25,26,27,28-tetra[(3-aminopropyl)oxy]-calix[4]arene 7

25,26,27,28-Tetra[(3-phthalimidopropyl)oxy]-calix[4]arene **6** (1.0 g, 0.85 mmol) and $NH_2NH_2 \cdot H_2O$ (0.83 g, 17 mmol) were solved in ethanol (50 mL), and this mixture was refluxed at 110–120 °C for 16 h. Then, the solvent was removed under reduced pressure. The residue was dissolved in CH_2Cl_2 (30 mL) and washed with a solution of NH_4OH (pH 9) (3 × 15 mL). The organic phase was separated, dried with $MgSO_4$, and evaporated to afford **7** as a pure solid. Yield 90%; mp 179–181 °C; FTIR: 3369 cm^{-1} (NH_2); ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.05 (d, 8H, $J = 7.8$ Hz, ArH), 6.80 (t, 4H, $J = 7.4$ Hz, ArH), 3.74 (s, 8H, $ArCH_2Ar$), 3.62 (t, 8H, $J = 6.8$ Hz, NCH_2), 2.64 (t, 8H, $J = 6.8$ Hz, OCH_2), 1.61 (p, 8H, $J = 6.8$ Hz, $OCH_2CH_2CH_2N$). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 156.6, 133.8, 129.8, 123.7, 68.4, 39.9, 37.9, 34.1. Calculated for $C_{40}H_{52}N_4O_4$: C, 73.59%; H, 8.03%; N, 8.58%. Found: C, 73.62%; H, 8.11%; N, 8.61%.

1,3-*Alternate*-25,26,27,28-tetra[(3-iminotriphenylaminepropyl)oxy]-calix[4]arene 8

A solution of 4-formyltriphenylamine (1.33 g, 6.20 mmol) and triethylamine (1 mL, excess) in methanol (20 mL) was added to a solution of 25,26,27,28-tetra[(3-aminopropyl)oxy]-calix[4]arene **7** (1.0 g, 1.53 mmol) in CH_2Cl_2 (30 mL). The mixture was refluxed for 24 h under nitrogen. After evaporation of the solvent, the solid residue was washed with cool methanol twice to remove unreacted 4-formyltriphenylamine and dried in vacuum. Yield 75%; mp 180–184 °C; FTIR: 1640 cm^{-1} (HC=N); ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 1.86 (p, 8H, $J = 6.6$ Hz, $CH_2CH_2CH_2$), 3.54 (t, 8H, $J = 6.6$ Hz, NCH_2), 3.66 (t, 8H, $J = 6.6$ Hz, OCH_2), 3.71 (s, 8H, $ArCH_2Ar$), 6.72 (t, 4H, $J = 7.2$ Hz, ArH), 7.03–7.12 (m, 40H, ArH), 7.24–7.28 (m, 16H, ArH), 7.57 (d, 8H, $J = 8.4$ Hz), 8.13 (s, 4H, CHN); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 160.44, 156.83, 150.17, 147.44, 134.04, 130.17, 130.00, 129.94, 129.60, 129.22, 126.53, 125.31, 123.81, 122.39, 119.57, 69.69, 58.57, 37.31, 31.83. Calculated for $C_{116}H_{104}N_8O_4$: C, 83.22%; H, 6.26%; N, 6.69%. Found: C, 83.31%; H, 6.34%; N, 6.76%.

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