Shape-Persistent Arylenevinylene Macrocycles (AVMs) Prepared via Acyclic Diene Metathesis Macrocyclization (ADMAC)

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SUPPORTING INFORMATION

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1. Materials and general methods

Reagents and solvents were purchased from commercial suppliers and used without further purification, unless otherwise indicated. Ether, tetrahydrofuran, toluene, CH_2Cl_2 and DMF are purified by MBRAUN solvent purification systems.

All reactions, except those performed in aqueous solvent, were conducted under dry nitrogen in oven dried glassware. Unless otherwise specified, solvents were evaporated using a rotary evaporator after workup. Unless otherwise specified, the purity of the compounds was ≥ 95 % based on ^{1}H NMR spectral integration.

Flash column chromatography was performed by using a 100-150 times weight excess of flash silica gel 32-63 μm from Dynamic Absorbants Inc. Fractions were analyzed by TLC using TLC silica gel F254 250 μm precoated-plates from Dynamic Absorbants Inc.

Analytical gel permeation chromatography (GPC) was performed using a Viscotek GPCmaxTM, a Viscotek Model 3580 Differential Refractive Index (RI) Detector, a Viscotek Model 3210 UV/VIS Detector and a set of two Viscotek Viscogel columns (7.8 × 30 cm, l-MBLMW-3078, and l-MBMMW-3078 columns) with THF as the eluent at 30 °C. The analytical GPC was calibrated using monodisperse polystyrene standards.

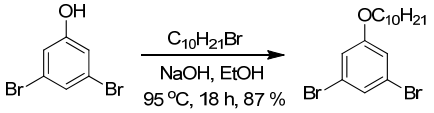
UV-vis absorption measurements were carried out with Agilent 8453 spectrophotometer and the emission measurements were obtained on a F-2500 Hitachi fluorescence spectrophotometer.

SEM images were recorded using a JSM-6480LV (LVSEM) at 5.0 kV. Sample was sputter-coated with gold prior to analysis.

Mass spectra were obtained on the Voyager-DE™ STR Biospectrometry Workstation using sinapic acid as the matrix.

NMR spectra were taken on Inova 400 and Inova 500 spectrometers. $CHCl_3$ (7.26 ppm) was used as internal references in ^{1}H NMR, and $CHCl_3$ (77.23 ppm) for ^{13}C NMR. ^{1}H NMR data were reported in order: chemical shift, multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet), number of protons, coupling constants (*J*, Hz).

2. Experimental procedures



1,3-Dibromo-5-decyloxy-benzene: The procedure reported by Kandre *et. al* was followed.[1] A mixture of 3,5-dibromophenol (1.0 g, 3.97 mmol) and 1-bromodecane (922 mg, 4.17 mmol), and NaOH (167 mg, 4.17 mmol) in EtOH (25 mL) was heated in a schlenk tube at 95 °C for 18 h. It was then allowed to cool to room temperature. Water (50 mL) was added and the product was extracted with diethyl ether (4 x 40 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, concentrated, and purified by flash column chromatography using hexane as the eluent to provide the product as a colorless oil (1.35 g, 87 %): ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 7.23 (1 H, t, *J* = 1.6 Hz), 6.99 (2 H, d, *J* = 1.6 Hz), 3.92 (2 H, t, *J* = 6.5 Hz), 1.92 – 1.70 (2 H,

[1] R. Kandre, K. Feldman, E. Meijer, E. H. Han, P. Smith and A. D. Schlüter, *Angew. Chem. Int. Ed.* 2007, **46**, 4956 –4959