meso-Octamethylcalix[4]pyrrole: an old yet new transmembrane ion-pair transporter†‡

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Electronic supplementary information

Synthesis of meso-octabutylcalix[4]pyrrole

A few drops of methane sulfonic acid were added to a solution of 5-nonanone (12.5 mL, 0.072 mol) and pyrrole (5 mL, 0.072 mol) in ethanol (75 mL) and heated to reflux for 3 hours. The resulting brown-black solution was reduced to approximately half its original volume using a rotary evaporator and then refrigerated overnight. This procedure yields a white crystalline solid in 6% yield, which was isolated by vacuum filtration. Impurities presumed to be N-confused by-products were removed by washing with methanol. The residue was dried under vacuum at 30–40 °C overnight to give a light beige solid. When necessary, this material was purified by flash chromatography over silica gel by eluting with CH₂Cl₂; under these conditions, the product moves with the solvent front.

¹H NMR (300 MHz, CD₂Cl₂) δ 6.95 (s, 4H), 5.91 (d, 8H), 2.1-0.9 (br, 72H)

¹³C NMR (75 MHz, CD₂Cl₂) δ 137.0, 104.6, 42.4, 36.3, 26.2, 23.5, 14.2.

IR (cm⁻¹) 3444, 2954, 2929, 2859, 1466, 1415, 1378, 1183, 1035, 756, 724, 664, 509, 497.

ESI MS (-ve mode) [m/z, (%)] M+Cl⁻ 800.0

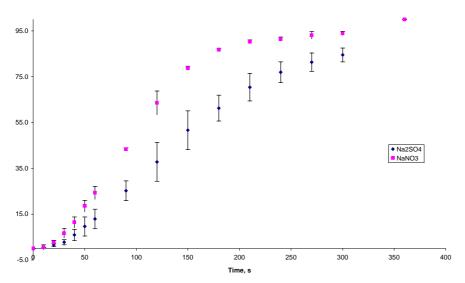


Figure S1 Chloride efflux promoted upon addition of **1** (2 % molar carrier to lipid) to unillamellar POPC vesicles loaded with 488 mM CsCl (■), 5mM phosphate buffer pH 7.2 dispersed in 488 mM NaNO₃ 5mM phosphate buffer pH 7.2 and unilamellar POPC vesicles loaded with 488 mM CsCl (●) 5mM phosphate buffer pH 7.2 dispersed in 145 mM Na₂SO₄ including error bars c.f. Figure 3 in communication.