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[16] All primers were provided by Sigma-Genosys, UK. The primers were provided as disulfides, and the thiolated oligonucleotides were prepared by reacting the commercial disulfides with 0.04 M threo-1,4-dimercapto-2,3-butandiol (DTT) for 16 h at room temperature followed by the purification of the reduced DNA on a NAP-10 column (Pharmacia).

[17] Quartz crystals, 9 MHz AT-cut sandwiched between two Au-electrodes (area 0.196 cm$^2$, roughness factor ca. 3.5) were used in the studies.

[18] The M13 mp8 (+) strand, nucleotides, biotinylated-dCTP and Klenow fragment were all provided by Sigma. Polymerization was performed in a 10 mM Tris buffer solution, pH 7.5, that contained 50 mM of KCl, 5 mM $MgCl_2$, and 20 U mL$^{-1}$ of the enzyme, dGTP, dATP, dTTP, dCTP, and biotinylated-dCTP (1:1:1:2/3:1/3, each base 1 mM).

[19] The avidin–alkaline phosphatase conjugate was coupled to the biotinylated surface by treating the electrodes or the quartz crystal with avidin–alkaline phosphatase conjugate (10 nmol mL$^{-1}$) in 0.1 M Tris buffer for 20 min. The resulting electrodes were treated with $2 \times 10^{-3}$ M 5-bromo-4-chloro-3-indolyl phosphate (Aldrich) in a Tris buffer for 20 min.

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[24] Enhanced avian reverse transcriptase product of Sigma.

[25] Yeast RNA of heterogeneous length of 2–7 kb (Boehringer Mannheim).

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# Cesium- and Rubidium-Selective Redox-Active Bis(calix[4]diquinone) Ionophores[**]

Philip R. A. Webber, George Z. Chen,
Michael G. B. Drew, and Paul D. Beer[*]

The synthesis of redox-active molecular receptors designed to selectively recognize and electrochemically sense charged or neutral guest species of biological and environmental importance is an area of intense current interest.[1] A number of research groups have incorporated redox-active transition metal and organic centers into a variety of macrocyclic structural frameworks based on crown ethers, cryptands, and calixarenes, and shown some of these systems to be selective and electrochemically responsive to the binding of metal cations, particularly lithium,[2] sodium,[3] and potassium.[4] However, the construction of redox-active ionophores for the selective recognition of the larger cesium and rubidium metal cations has not, to our knowledge, been reported. This situation is surprising in view of the environmental concern for monitoring radioactive cesium in nuclear waste solutions[5] and the potential use of rubidium isotopes in radiopharmaceutical reagents.[6] We report here the synthesis, coordination, and electrochemical investigations of novel bis(calix[4]-diquinone) receptors $L^1$ and $L^2$, and demonstrate their remarkable ability to selectively complex and electrochemically sense cesium and rubidium cations.

Reaction of a solution of *p-tert*-butylcalix[4]arene (**1**) in acetonitrile with propane-1,3-ditosylate, 1,4-dibromobutane, or 1,5-dibromopentane in the presence of potassium carbonate gave the bis(calix[4]arene) derivatives **2**, **3**, and **4** in 30, 23, and 26% yields, respectively (Scheme 1). Oxidation of these compounds with $Tl(OCOCF_3)_3$ in trifluoroacetic acid[4a, 7] gave the new bis(calix[4]diquinone) ionophores $L^1$, $L^2$, and $L^3$ in respective yields of 28, 10, and 9% after column chromatography and recrystallization.[8]

Electrospray mass spectrometry (ES-MS) competition experiments gave the first qualitative indication that $L^1$ and $L^2$ displayed notable selectivity preferences for $Cs^+$ and $Rb^+$ ions. The electrospray mass spectra of equimolar Group 1 metal iodides in the presence of solutions of the tetraquinone ligands in DMSO revealed the most intense peaks occurred at

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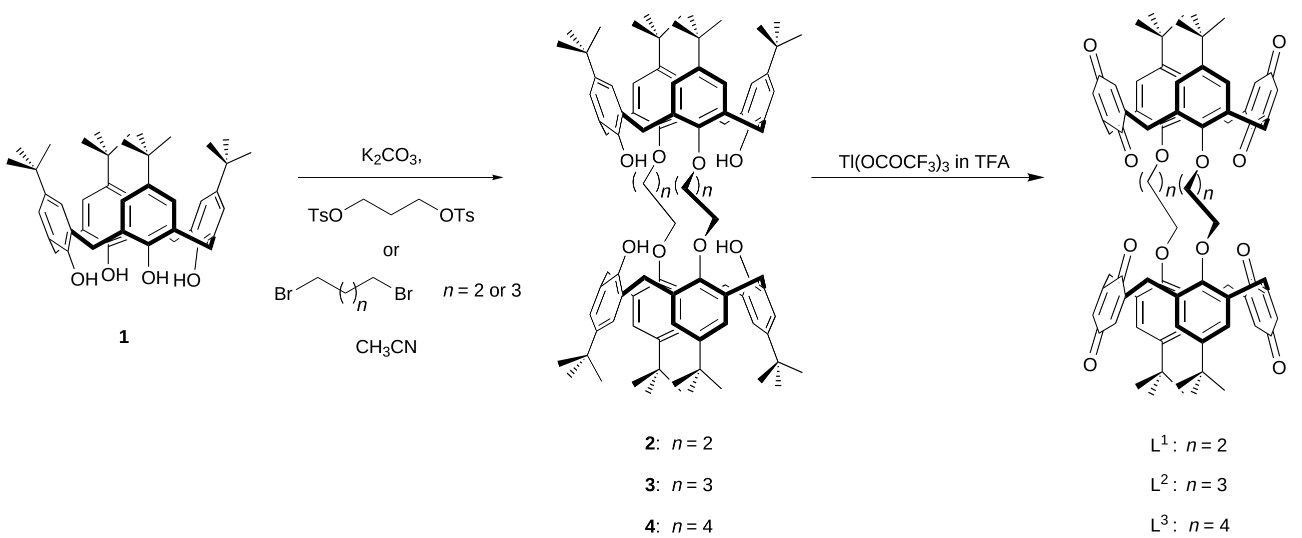

Scheme 1. Synthesis of the calix[4]diquinones. Ts = toluene-4-sulfonyl; TFA = trifluoroacetic acid.

*m/z* values of 1293.3 $[L^1+Rb]^+$ and 1369.9 $[L^2+Cs]^+$. In contrast, analogous competition ES-MS experiments with $L^3$ suggested only weak binding of Group 1 metal cations in DMSO solutions. UV/Vis titrations were undertaken on $L^1$ and $L^2$ in a mixture of DMSO and $H_2O$ (99:1) by monitoring the perturbation of the $n \rightarrow \pi^*$ electronic transition of the quinone moiety on addition of the cationic Group 1 metal guest. The stability constants for 1:1 complexes were then calculated using the Specfit program.[9] The data obtained (Table 1) reveals that $L^1$ forms a very stable complex with $Rb^+$ ions and exhibits the selectivity trend $Rb^+ > Cs^+ > K^+ \gg Na^+$. In contrast, $L^2$ displays a remarkable high selectivity for $Cs^+$ ions;[10] in fact in this competitive aqueous DMSO mixture $L^2$ does not form complexes with either $K^+$ or $Na^+$ ions (Table 1). This very high $Cs^+$:$Na^+$ selectivity preference is of real significance for the separation of radioactive cesium nuclear waste, as such solutions typically contain $Na^+$ ions at high concentrations.[5] It is noteworthy that the qualitative selectivity trends obtained from the ES-MS competition experiments are in agreement with the stability constant data and shows that the length of the bridging methylene chain between the two calix[4]diquinone moieties crucially dictates the selectivity and strength of binding for Group 1 metal cations.

Table 1. Stability constant data for the alkali metal complexes.

| | $K$ [$M^{-1}$][a] | |
|---|---|---|
| | $L^1$ | $L^2$ |
| $Na^+$ | –[b] | –[b] |
| $K^+$ | $7.9 \times 10^3$ | –[b] |
| $Rb^+$ | $6.3 \times 10^4$ | 40 |
| $Cs^+$ | $1.3 \times 10^4$ | $1.6 \times 10^3$ |

[a] Determined in DMSO:$H_2O$ (99:1) at 298 K, errors are estimated to be ≤5%. The alkali metal cations were added as their perchlorate salts. [b] No evidence of complexation was observed.

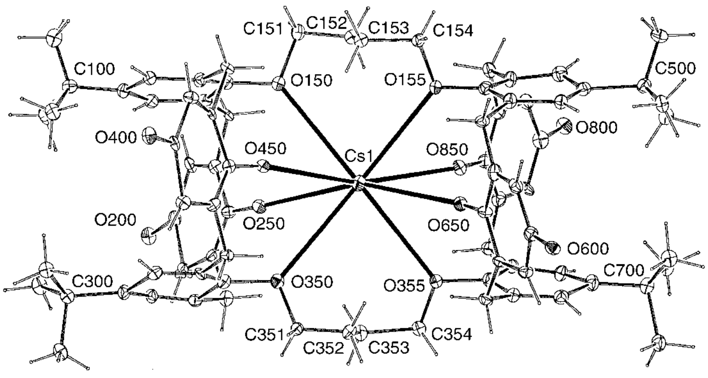

Figure 1. The structure of $[CsL^2]^+$ with ellipsoids at 10% probability.

The cesium complex of $L^2$ has been characterized by X-ray crystallography (Figure 1). The cation structure has approximate twofold symmetry. Both calix[4]diquinones have the well-known $C_2$ flattened-cone conformation. Thus, the phenyl rings (1, 3, 5, and 7) are almost perpendicular to the plane of the methylene rings, intersecting at angles of 87.5, 86.5, 89.1, and 84.7°, respectively, while the four diquinone rings (2, 4, 6, and 8) intersect at angles of 35.0, 35.0, 29.8, and 32.2°, respectively. The torsion angles for atoms C16-O150-C151-C152-C153-C154-O155-C156 in the butane link between the two oxygen atoms of the calixdiquinones show a *trans*, *−gauche*, *trans*, *−gauche*, *trans* conformation around the five central bonds, respectively, which allows bidentate coordination to the metal center. The cesium atom is bonded to the eight oxygen atoms bordering the central cavity. The shortest Cs–O distances are to the four diquinone oxygen atoms (3.224(8)–3.292(8) Å) with longer distances to the four ethereal oxygen atoms (3.364(9)–3.530(10) Å). In addition, the Cs atom forms two strong bonds to adjacent oxygen atoms from the diquinone oxygen atoms at the top rim of two neighboring molecules (O200: 3.226(8) ($-x$, $-y$, $-z$) and O400: 3.287(8) Å ($-x$, $1-y$, $-z$)), thus setting up a 1D polymeric chain parallel to the $y$ axis (Figure 2).

A preliminary electrochemical investigation of the recognition properties of $L^1$ and $L^2$ for Group 1 metal ions was performed in a mixture of dichloromethane and acetonitrile (4:1) by using cyclic voltammetry (CV) and square-wave

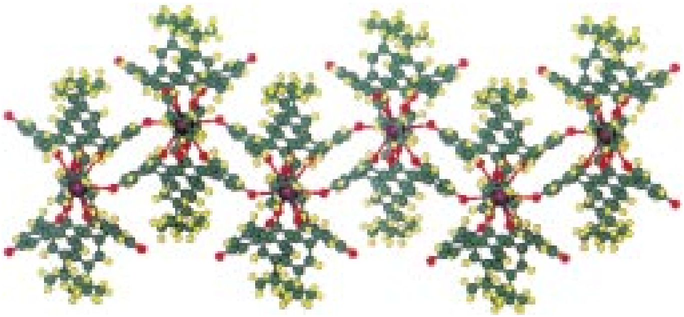

Figure 2. The structure of $[CsL^2]^+$ showing the polymeric chain in the *b* direction; cesium: purple, carbon: green, hydrogen: yellow, oxygen: red.

Table 2. Electrochemical data[a] in $CH_2Cl_2$:$CH_3CN$ (4:1).

| | $L^1$ | $L^2$ |
|---|---|---|
| $E_{1/2}$ [V][b] | – 1.16 | – 1.18 |
| $E_{pc}$ [V][c] | – 1.71 | – 1.79 |
| $\Delta E$ ($Na^+$) [mV][d] | 440 | 500 |
| | 290 | 440 |
| $\Delta E$ ($K^+$) [mV][d] | 360 | 440 |
| | 210 | 370 |
| $\Delta E$ ($Rb^+$) [mV][d] | 350 | 330 |
| | 210 | 270 |
| $\Delta E$ ($Cs^+$) [mV][d] | 340 | 310 |
| | 210 | 260 |

[a] 0.1M $Bu_4NBF_4$ was added as the supporting electrolyte. The concentration of the receptor in solution was approximately $1\times10^{-3}$M. [b] $E_{1/2}$ values are the averages of the cathodic and anodic peak potentials of the first wave couple referenced to a Ag/$Ag^+$ electrode at 298 K. [c] $E_{pc}$ is the cathodic peak potential of the second wave, which is irreversible. [d] Anodic shifts of the reduction waves relative to the quasireversible first reduction wave of the free receptor on addition of up to five equivalents of alkali metal cations as their perchlorate salts.

voltammetry (SWV, Figure 3). The results, although complicated by protonation of reduced radical anion and dianionic species,[4a, 11, 12] are summarized in Table 1. Both receptors exhibit essentially two broad redox waves, which implies that there is multielectron transfer. The two redox waves correspond to a quasi-reversible reduction and an irreversible redox process which occurs at a more cathodic potential (Table 2). These CV features are very similar to those of previously reported calix[4]diquinones.[4a, 11, 12] By taking into account the known voltammetric behavior of a calix[4]diquinone, which can accept a total of four electrons by sequential two-electron reduction processes, the first redox wave may be attributed to a four-electron reduction process in which a one-electron transfer takes place to each of the quinone moieties present in the ligand to form radical anions. The second, more cathodic, irreversible reduction is consistent with another four-electron reduction process which leads to the formation of dianions. It has been suggested that the irreversibility of the second wave could be the result of the formation of insoluble hydroquinone species.[12] The addition of Group 1 metal cations to electrochemical solutions of $L^1$ and $L^2$ led in all cases to the evolution of two new redox waves at potentials that were substantially positive-shifted relative to wave 1, which diminished in current height and shifted anodically concomitant with the growth of the new waves (Figure 3). Surprisingly the growth of the new waves ceased after the addition of one equivalent of metal cation, while wave 1 was still at a significant current height. Wave 1 cannot be simply ascribed to uncomplexed ligand because of its positive shift in reduction potential. Instead the observed metal cation induced electrochemical behavior suggests the metal cation is bound in an unsymmetrical manner, in closer proximity to one of the two calix[4]diquinone moieties. Although the solid-state structure of $[CsL^2]^+$ (Figure 1) shows the cesium cation is symmetrically located in the center of the ligand, the movement of the complexed cation may occur on reduction, during which electron transfer to one of the calix[4]diquinone groups proceeds earlier in real time than to the second calix[4]diquinone group.[13] Thus, the resulting negative charge on one of these redox centers could induce movement of the metal cation within the tetraquinone–polyether binding site.[14, 15] The alkali metal cation induced anodic shift values relative to the first reduction wave of the respective receptor (Table 2) are all substantial in magnitude, with $Na^+$ ions producing the greatest magnitude of shift ($\Delta E = 500$ mV). It is noteworthy that electrochemical competition experiments with $L^2$ in a mixture of $CH_2Cl_2$ and $CH_3CN$ (4:1) in the presence of tenfold excess amounts of $Na^+$ and $K^+$ ions did not affect the receptor's selective redox response to $Cs^+$ ions.

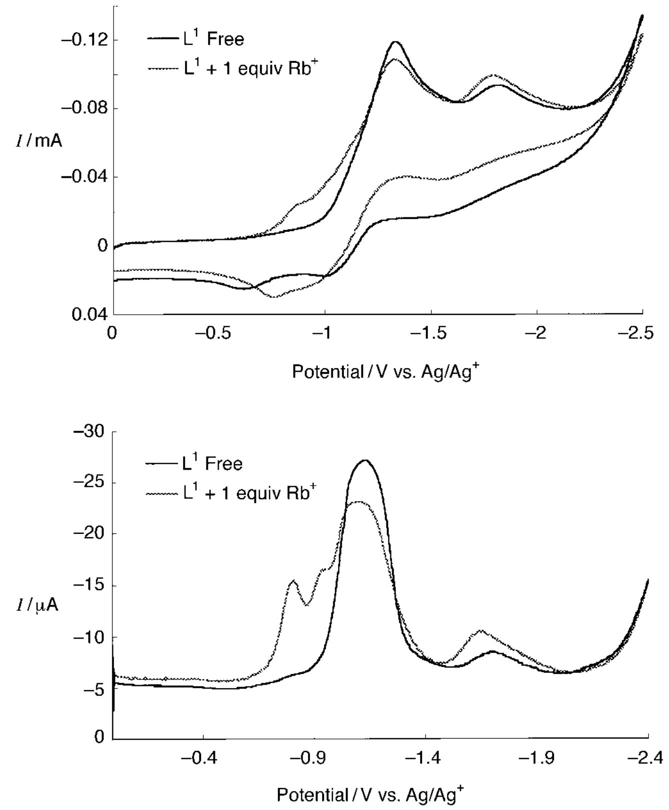

Figure 3. CV (top) and SWV (bottom) of $L^1$ and $L^1$ plus one equivalent of $Rb^+$ in $CH_2Cl_2$:$CH_3CN$ (4:1). The anodic peaks at – 1.6 V and – 1.0 V are coupled with the cathodic peaks at – 1.7 V and – 1.3 V, respectively. It is noteworthy that the shapes of the two-wave couples are affected by the protonation of the reduced radical anion and dianion. The current wave at approximately – 0.8 V on the reverse scan has also been observed in the CV spectra of other calix[4]diquinones, and was ascribed to incompletely protonated dianions (see ref. [11]). This argument is also supported by the fact that the current wave at – 0.8 V is not observed when a metal cation is complexed.

In summary these new bis(calix[4]diquinone) receptors represent a new design of redox-active ionophore which depend upon the length of the bridging methylene chain between the two calix[4]diquinone moieties. Their unique topological cavities can exhibit impressive selectivity preferences and novel electrochemical recognition properties towards the larger alkali metal cations $Cs^+$ and $Rb^+$.

## *Experimental Section*

Experimental details are given in the Supporting Information. Crystal data for $[CsL^2]ClO_4$: $C_{82}H_{93}ClCsO_{19.5}$, $M_r = 1558.92$, monoclinic, space group $P2_1/n$, $a = 11.869(15)$, $b = 18.337(25)$, $c = 40.992(44)$ Å, $\beta = 91.00(1)^\circ$, $V = 8920$ Å$^3$, $Z = 4$, $\rho_{calcd} = 1.161$ Mg m$^{-3}$, $\mu = 0.506$ mm$^{-1}$. Intensity data was collected with $Mo_{K\alpha}$ radiation using the *MAR*research Image Plate System. The crystal was positioned 90 mm from the Image Plate. 100 frames were measured at 2° intervals with a counting time of 10 mins to give 8091 independent reflections. Data analysis was carried out with the XDS program.[16] The structure was solved by using direct methods with the SHELX86 program.[17] In the cesium complex of $L^2$ there are two independent perchlorate anions with occupancies refined to 0.56, 0.44 and two pairs of overlapping methanol molecules each with common oxygen atoms and occupancies refined to 0.49, 0.51 and 0.49, 0.51, respectively, and two water molecules with occupancies of 1.0 and 0.5. The non-hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms were included in geometric positions and given thermal parameters equivalent to 1.2 times those of the atom to which they were attached. The structure was refined on $F^2$ using SHELXL.[18] The final $R$ values for the cesium complex of $L^2$ were $R_1 = 0.0891$, $wR_2 = 0.2398$ for data with $I > 2\sigma(I)$. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-157775 and CCDC-157776. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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[13] Variable-temperature $^1H$ NMR experiments with $L^2$ and $Cs^+$ ions carried out using the same solvent mixture of dichloromethane and acetonitrile (4:1) and also in DMSO gave no evidence for the existence of an unsymmetrical complex on the NMR time scale. These observations suggest electron transfer is necessary for this unsymmetrical complex to form.

[14] For redox-active receptors where through-space electrostatic interactions dominate the potential shift, $\Delta E$ (mV) can be calculated using the equation $\Delta E = (Q_{redox}e/4p\varepsilon\varepsilon_0 l)(z/r)$, where $Q_{redox} = ne$, the charge variation in the redox center upon electron transfer; $e$ = charge on an electron; $n$ = number of electrons transferred to or from the redox center; $\varepsilon_0$ = permittivity of a vacuum; $\varepsilon$ = relative permittivity of the solvent; $l$ = distance between the redox center and the complexed cation; $z$ = valence of the complexed cation; and $r$ = radius of complexed cation. (See: Z. Chen, A. J. Pilgrim, P. D. Beer, *J. Electroanal. Chem.* **1998**, *444*, 209.) For 4:1 $CH_2Cl_2:CH_3CN$ $\varepsilon = 14.6$, $r(Cs^+) = 1.74$ Å, average $Cs^+{-}O$ distance from crystal structure = 3.258 Å, $\Delta E = 173$ mV. (If the distance is taken between the centers of the complexed cation and quinone redox center, the value of $\Delta E$ would be smaller in magnitude.) This value is much smaller than the experimentally observed value of 260 mV (Table 2), which suggests the $Cs^+$ ion is bound closer to one of the two calix[4]diquinone redox centers.

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# Cyclic Hexamer with a Cubic Cavity: Crystal Structure of $[\{Rh(6\text{-Purinethione Ribosido})(Cp^*)\}_6](CF_3SO_3)_6$

Kazuaki Yamanari,* Shiori Yamamoto, Rie Ito, Yoshihiko Kushi, Akira Fuyuhiro, Naoko Kubota, Tsuyoshi Fukuo, and Ryuichi Arakawa

The metal–ion binding capability of $N^9$-substituted nucleobases such as 9-alkyladenine (R-Hade) and adenosine (Hado) have been studied intensively in the past two decades.[1] Some cyclic polynuclear metal complexes that include such nucleobases have been synthesized recently based on self-assembling reactions.[2] Crystal structures have been reported for $[\{Rh(Me\text{-}ade)(Cp^*)\}_3](CF_3SO_3)_3$ ($Cp^* = \eta^5\text{-}C_5Me_5$),[3]

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