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Parallels in Cation and Anion Coordination: A New Class of Cascade Complexes**

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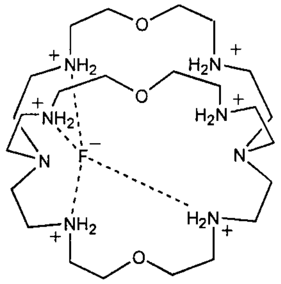
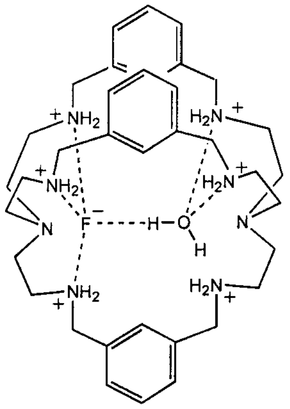
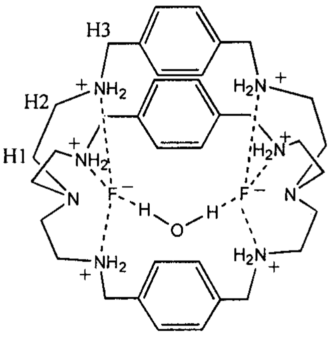
For a number of years it has been speculated that azacryptands capable of binding two metal ions could also serve as hosts for two discrete anions. In the early 1980s Lehn and co-workers proposed that a simple bicyclic azacryptand (bis-tren) derived from two tren (tris(2-aminoethyl)amine) units might be capable of encapsulating a bifluoride ion.[1] Although not considered as "discrete" ions in that regard, bifluoride does consist of two fluoride ions linked by a hydrogen bridge. However, a crystal structure revealed only a single fluoride ion sitting to one side of the cavity (**A**). In 1998 we observed ditopic anion binding behavior, namely, two discrete encapsulated nitrate ions, for a related azacryptand, MEAcryp.[2, 3] Our subsequent attempt to sequester either a bifluoride ion or two discrete fluoride ions in the MEAcryp cavity was not successful, although crystallographic results did indicate two residents in the cavity: a molecule of water and a fluoride ion (**B**).[4, 5]



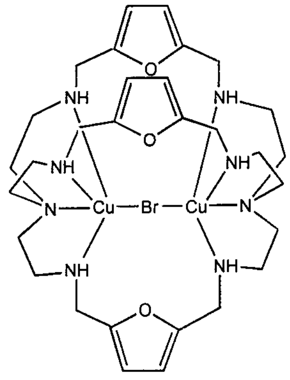
bis-tren(F)
A



MEAcryp(F)(H$_2$O)
B



PEAcryp(F)$_2$(H$_2$O)
C



FuEAcryp(Cu)$_2$(Br)
D

By very slightly enlarging the cavity size with a *p*-xylyl spacer to give PEAcryp (**L**),[2] we have at last succeeded in capturing two fluoride ions inside the cavity (**C**), to provide the first example of an azacryptand with two encapsulated fluoride ions. Rather than being linked by a single hydrogen atom, as for a bifluoride ion (F-H-F$^-$), a water molecule bridges the two halide ions (F$\cdots$H-O-H$\cdots$F^{2-}). This additional structural feature adds another dimension to the finding, thus making this complex the first example of an "anion-based cascade complex," where two spherical anions play the topological role of the two metal ions in traditional cascade complexes. These findings serve to expand the

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Supporting information for this article is available on the WWW under http://www.angewandte.org or from the author.

increasing number of examples of analogies between traditional transition metal coordination chemistry and anion coordination chemistry, where hydrogen bonding in the latter replaces lone-pair coordination in the former.

Cascade complexes in ditopic aza macrocycles and cryptands are well-documented and were first termed as such over two decades ago.[6] Normally these complexes consist of monocyclic or bicyclic ligands with two encapsulated metal ions and a bridging anion. More recently, researchers have advanced the field significantly by identifying more readily accessible macrocycles and cryptands derived from Schiff base condensations between amines and aromatic or heterocyclic dialdehydes, followed by simple reductions of the resulting imines to amines.[7] These ligands form similar types of cascade complexes with metal ions, as in the recently reported dicopper cascade complex of a bis-tren cryptand with a furan spacer (**D**).[8]

In this first example of an anion-based cascade complex, $[H_6\mathbf{L}(F)_2(H_2O)][SiF_6]_2 \cdot 12\,H_2O$ (**1**), **L** is hexaprotonated, and is surrounded by two external SiF_6^{2-} counterions and twelve molecules of water.[9–12] Thus, the only fluoride ions in the structure are in the cavity of **L**. Figure 1 shows views looking into the cavity and down the threefold axis (a and b, respectively). The view in Figure 1b shows that the two fluoride ions reside almost in the center of the cavity, with the water bridge somewhat to the outside: F1 is 0.124 Å and F2 is 0.229 Å from a line between N1 and N16. Each fluoride ion is coordinated in a very distorted tetrahedron through hydrogen-bonding interactions to three protonated secondary amines of a given tren unit and the bridging water molecule (Table 1, Figure 1c). The distortion arises from the fact that the water molecule lies between two of the three cryptand "arms," the N4 ··· N13 and N19 ··· N28 bridges, which results in very large O-F-N angles for the farside bridge (N33 ··· N42) of 152.0 and 140.6°.

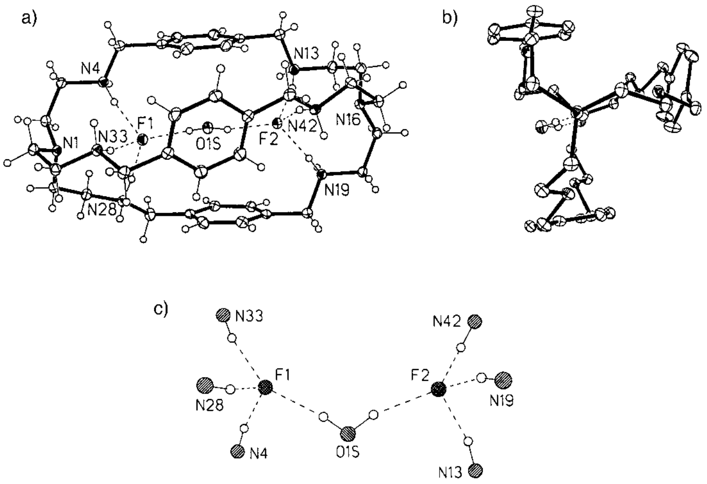

Figure 1. Views of $[H_6\mathbf{L}(F)_2(H_2O)]^{4+}$: a) side view showing the water cascade; b) view down the pseudo-threefold axis; c) view showing only the coordination of the fluoride ions. The external anions and the water molecules of crystallization are omitted for clarity.

Table 1. Interatomic distances and angles for hydrogen-bonding interactions of the encapsulated fluoride ions for **1**.

Atoms	Distance [Å]	Atoms	Distance [Å]
F1 ··· N4	2.6703(14)	F(2) ··· N(13)	2.6538(14)
F1 ··· N28	2.6042(14)	F(2) ··· N(19)	2.6820(14)
F1 ··· N33	2.7237(12)	F(2) ··· N(42)	2.6100(14)
F1 ··· O1S	2.7090(13)	F(2) ··· O(1S)	2.7168(13)
Atoms	**Angle [°]**	**Atoms**	**Angle [°]**
N4 ··· F1 ··· N28	111.5	N13 ··· F2 ··· N19	97.4
N4 ··· F1 ··· N33	96.7	N13 ··· F2 ··· N42	107.2
N28 ··· F1 ··· N33	97.1	N19 ··· F2 ··· N42	100.3
O1S ··· F1 ··· N4	99.0	O1S ··· F2 ··· N13	91.6
O1S ··· F1 ··· N28	98.5	O1S ··· F2 ··· N19	111.4
O1S ··· F1 ··· N33	152.0	O1S ··· F2 ··· N42	140.6

The fluoride ions are almost equidistant from their respective bridgehead amines, at distances (2.994 and 3.002 Å) which are slightly less than the sum of the van der Waals radii of nitrogen and fluorine atoms (3.02 Å).[13] The hydrogen bonds from the fluoride ions to the amines range from 2.6 to just above 2.7 Å, with slightly longer hydrogen bonds to the water cascade. The fluoride ··· fluoride distance is 4.736 Å. The distance between the bridgehead nitrogen atoms (N1 and N16) of **L** is 10.717 Å, which results in an elongated ellipsoidal shape for the cryptand, and is probably the result of the two "guests" in the cavity.

An attempt to isolate a mixed fluoride/chloride complex resulted in a second cryptand structure in which two fluoride ions were found inside the cavity, $[H_6\mathbf{L}(F)_2(H_2O)][Cl]_2[SiF_6] \cdot 9\,H_2O$ (**2**). Again the fluoride ions are bridged by a water molecule, but two chloride ions and a single SiF_6^{2-} ion serve as counterions, with nine additional water molecules of crystallization.[14] The isolation of an analogous structure in the presence of competing anions indicates that the fluoride cascade complex represents a preferred structural pattern for fluoride ions with PEAcryp (**L**).

Protonation constants for **L** and binding constants for fluoride ions with **L** were determined by potentiometric methods, and the latter verified by NMR titrations. Although protonation constants for **L** were determined previously by others using Et_4NClO_4 as the electrolyte,[15] we recalculated the constants using NaTsO (Ts = toluene-4-sulfonyl) as the electrolyte of choice for anion binding studies, as the bulky TsO^- ion is presumed to provide minimal competition for binding. The two series of pK_a values were in good agreement, with the exception of the fifth and sixth protonation steps, for which our values (5.80(4) and 5.68(2)) were almost one unit less than those previously reported (6.7(1) and 6.52(9)).[15] Earlier studies on the influence of various electrolytes in these systems indicated that higher pK_a values in the lower pH region are observed in the presence of anions with binding capabilities, as would be the case for perchlorate.[16]

Binding constants were determined by both potentiometric and NMR methods. The potentiometric results indicated two viable models, one with an $[H_6LF]^{5+}$ species and the other with an $[H_6LF_2]^{4+}$ species at low pH values (maximizing at about pH 4), but not a model where both occurred simultaneously. The system is very complex, however, as a consequence of the proximity of the fifth and sixth protonation constants, and so the actual species present at lower pH values become difficult to define unambiguously. Since the dinuclear results gave better statistics and were chemically plausible, these are taken as the best model (Figure 2). Furthermore, the ditopic model is in agreement with the crystal structure analysis. The results indicated $K_{[H_3LF]/[H_3L][F]} = 3.16(2)$, $\log K_{[H_4LF]/[H_4L][F]} = 3.24(4)$, $\log K_{[H_5LF]/[H_5L][F]} = 3.96(2)$, and $\log K_{[H_6LF]/[H_2L][F]^2} = 6.54(5)$. The NMR results compare favorably with the potentiometric results and yielded $\log K_a = 3.13(5)$ for a 1:1 complex at pD 5.0, where potentiometric data indicate that the complex is a mixture of penta- and hexaprotonated receptors. The NMR titration of **L** with NaF at pD 5.0 gave the best fit for a 1:1 NaF:**L** ratio (Figure 3), in agreement with a Job's plot performed at pD 5.0, which indicated a maximum $\Delta\delta$ value at $0.6 = [\mathbf{L}]/\{[\mathbf{L}] + [F^-]\}$. These results, taken together, tend to indicate that a dinuclear complex is not the primary species in solution, although it may be present at lower pH values.

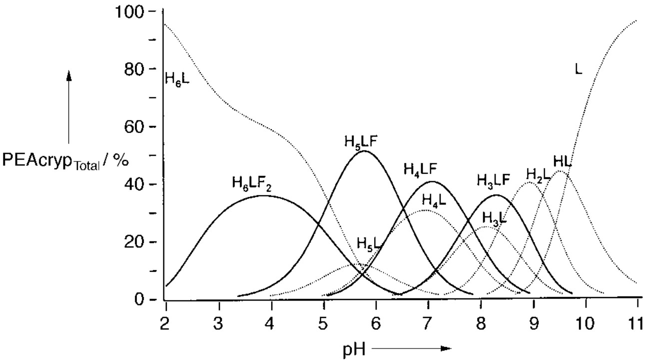

Figure 2. NMR titration curves for the three aliphatic signals of **L**, ΔH1 (▲), ΔH2 (●), and ΔH3 (■) with NaF in D_2O at 25 °C and pD = 5.0 ± 0.1.

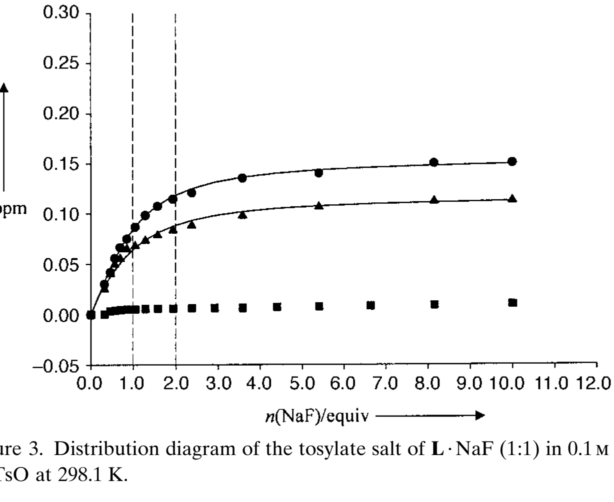

Figure 3. Distribution diagram of the tosylate salt of **L**·NaF (1:1) in 0.1 M NaTsO at 298.1 K.

^{19}F NMR spectroscopy was also used to probe the solution structure of the complex (Figure 4). Two signals are evident at pH 5.0: one at $\delta = -78$ ppm, and one at $\delta = -113$ ppm. The former is assigned to an internal fluoride ion, while the latter is characteristic of a solvated or external fluoride ion.[4] The signal at $\delta = -78$ ppm is still sharp at pH 5.5, but quite broad at pH 6.5 and 7.0, and is completely absent at pH 7.5. This observation contrasts with that found for the MEAcryp complex (**B**), where only an internally bound fluoride ion was observed between pH 5.0 and 6.5.[4]

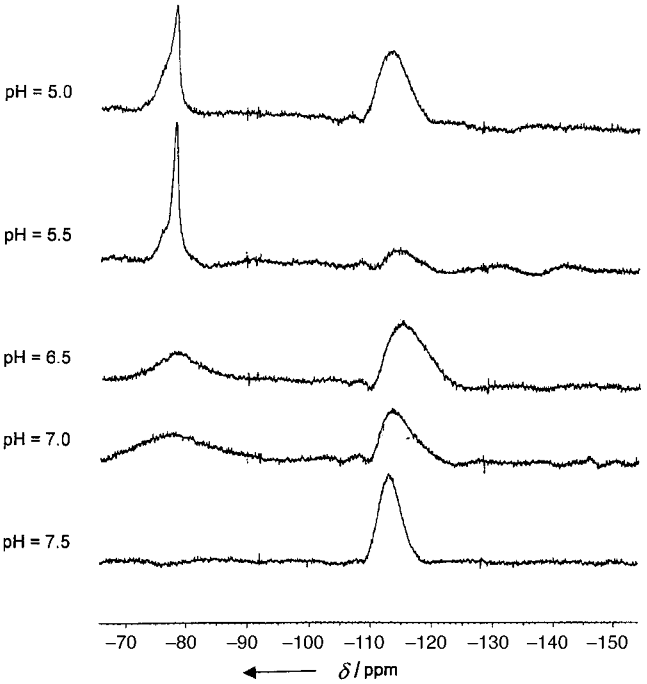

Figure 4. ^{19}F NMR spectra of NaF:PEAcryp·6Ts (10 mM) in 0.1 M KTs at −25 °C in DMSO:H_2O (40:60).

In conclusion, this structure represents the first example of two fluoride ions within an azacryptand cavity. Additionally, a new class of cascade complexes with water as the cascade has been identified, where anions play the topological role of the metal ions. This finding further expands on the analogies between traditional transition metal coordination chemistry and anion coordination chemistry, where hydrogen bonds in the latter fill the role of the coordinate covalent bonds in the former.

Experimental Section

1: PEACryp (**L**) was synthesized according to previously published methods and isolated as the free base.[5] The fluoride complex was obtained by titrating a solution of **L** in methanol with a 48% solution of HF to pH 2.0. A white crystalline powder formed immediately, which was recrystallized from a mixture of isopropanol and water to yield X-ray quality colorless plates. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): $\delta = 2.87$ (t, 12H, NCH_2), 3.31 (t, 12H, NCH_2CH_2), 4.25 (s, 12H, $ArCH_2$), 7.43 ppm (d, 12H, ArH); ^{13}C NMR (125 MHz, $CDCl_3$, TMS): $\delta = 45.0$ (NCH_2), 48.8 (NCH_2CH_2), 51.0 ($ArCH_2$), 129.8 (C_{Ar}), 131.9 ppm (CH_{Ar}); FAB-MS: m/z 599 $[HL]^+$, 619 $[H_2L^{2+}+F^-]$, 639 $[H_3L^{3+}+2F^-]$. Elemental analysis calcd for ($C_{36}H_{78}N_8F_{14}O_9Si_2$): C 39.70, H 7.21, N 10.28; found: C 39.77, H 7.04, N 10.23.

Potentiometric measurements: The potentiometric titrations were carried out in 0.1 M NaOTs at 298.1 ± 0.1 K. Electromotive force (EMF) data was obtained using an Orion 81–02 electrode. The electrode was calibrated as a hydrogen-ion concentration probe by titration of previously standardized amounts of HCl with CO_2-free NaOH solutions and determining the equivalent point by the Gran's method, which gives the standard potential E^o and the ionic product of water ($pK_w = 13.73(1)$). The computer program SUPERQUAD[17] was used to calculate the protonation and stability constants. The titration curves for each system were obtained from about 100 experimental points corresponding to at least three measurements taken along the pH range 2.0–11.0. The concentrations of both ligand and fluoride ions were 1 to 2 mM. The protonation constant of fluoride in water is: $\log K_{HF/F} = 3.15$.

NMR Measurements: ^{1}H NMR spectra were recorded on a Bruker AM500 spectrometer at 500 MHz. Binding constants were obtained by NMR titrations of **L** with fluoride from 25 measurements in D_2O at $pD = 5.0 \pm 0.1$. Initial concentrations were $[\mathbf{L}]^0 = 2$ mM and titrations were performed using aliquots from a 20 mM stock solution of NaF. A solution of the sodium salt of [2,2,3,3-D_4]-3-(trimethylsilyl)propionic acid (TPS) in D_2O in a capillary tube was used as an external reference. The pD value was adjusted with a concentrated solution of TsOH and NaOD in D_2O. All spectra were recorded at room temperature. The association constants K_s were calculated by fitting f to δ_{obs} (consisting of several independent NMR signals) with a 1:1 association model using Sigma Plot software. Equations (1) and (2) were used, where **L** is the ligand and A^- is the anion, and the error limit in K is less than 10%:

$$c = ([A^-]^0 + [\mathbf{L}]^0 + 1/K_s - \{([A^-]^0 + [\mathbf{L}]^0 + 1/K_s)^2 - 4[\mathbf{L}]^0[A^-]^0\}^{1/2})/2 \quad (1)$$

$$f = (\delta_{LA} - \delta_L)c/[\mathbf{L}]^0 + \delta_L \quad (2)$$

The Job's plot was performed by examining different concentration ratios of **L** and NaF in D_2O at $pD = 5.0 \pm 0.1$, while maintaining the total concentration of the ligand plus NaF at 10 mM. The pD value was adjusted with a concentrated solution of TsOH and NaOD in D_2O. NMR measurements were recorded at room temperature.

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[2] Nomenclature: M = *m*-xylyl, P = *p*-xylyl, and Fu = furan spacers; E = ethyl links within the tren unit between amine groups; A = amine; cryp = cryptand.

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[9] X-ray data for **1**: A crystal of dimensions 0.64 × 0.42 × 0.21 mm was selected for structural analysis. Crystal data: $M_r = 1161.31$; triclinic space group, $P\bar{1}$, $a = 9.8302(11)$, $b = 15.9973(19)$, $c = 17.417(2)$ Å, $\alpha = 84.404(3)$, $\beta = 89.959(3)$, $\gamma = 77.098(2)^\circ$, $V = 2656.5(5)$ Å^3, $Z = 2$, $\rho_{calcd} = 1.452$ g cm^{-1}, $F(000) = 1232$. A total of 12226 observed reflections ($I > 2\sigma(I)$) were collected. The refinement converged at $R1$(F, observed data) = 0.0443, and $wR2$ (F^2, all data) = 0.1189, and GOF = 0.974. Diffraction data were collected using a Bruker SMART APEX CCD area detector mounted on a Bruker D8 goniometer using graphite-monochromated $Mo_{K\alpha}$ radiation ($\lambda = 0.71073$ Å) at 100(2) K.[8] Intensity data were measured as a series of ω and θ oscillation frames each of 0.3° for times of 5 s per frame. The detector was operated in a 512 × 512 mode and was positioned 5.04 cm from the sample. Coverage of unique data was close to 99.4% complete to 26.00° in θ. Cell parameters were determined from a least-squares fit of 9504 peaks in the range $2.40 < \theta < 30.58^\circ$. Virtually no decay was observed, based on data obtained for a number of peaks monitored at both the beginning and end of data collection. The data were corrected for absorption by the semi-empirical method.[9] Lorentz and polarization corrections were applied. The data were merged to form a set of 15608 independent data with $R_{int} = 0.0248$. The space group was determined by statistical tests, and the structure was solved by direct methods and refined by full-matrix least-squares methods on F^2.[10] Hydrogen atom positions were initially determined by geometry and refined by a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. CCDC-172118 (**1**) and CCDC-172119 (**2**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

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[14] Synthesis, characterization, and structural results for **2** are given in the Supporting Information.

[15] Protonation constants for PEAcryp have been reported as $\log K_1 = 9.6(1)$, $\log K_2 = 9.00(4)$, $\log K_3 = 8.62(8)$, $\log K_4 = 7.3(1)$, $\log K_5 = 6.7(1)$, and $\log K_6 = 6.52(9)$ using Et_4NClO_4 as the electrolyte: F. Arnaud-New, S. Fuangswasdi, B. Maubert, J. Nelson, V. McKee, *Inorg. Chem.* **2000**, *39*, 573–579.

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Stereoselective Formation of Quaternary Carbon Centers: Alkylation of α,α-Disubstituted Amide Enolates**

Jeffrey M. Manthorpe and James L. Gleason*

The stereoselective formation of quaternary carbon centers is one of the most challenging tasks in organic chemistry and can only be achieved using methods which employ some form of carbon–carbon bond forming reaction.[1] One of the most straightforward methods for the formation of carbon–carbon bonds is the alkylation of an enolate with an alkyl halide and,

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