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Comparison of the structural, electrochemical, and spectroscopic properties of two cryptates of trivalent uranium†

D. Nuwangi Kulasekara,[a] Matthew D. Bailey,[a] Cassandra L. Ward[b] and Matthew J. Allen *[a]

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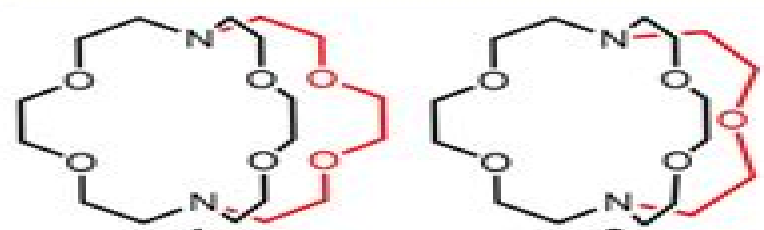
We describe a study of the influence of cryptand denticity on the structural, electronic, and electrochemical properties of U^{III}-containing cryptates. Two cryptands (2.2.2 and 2.2.1) are reported. The cryptand with the smaller denticity leads to negative electrochemical potentials and shorter bond lengths that are consistent with a better fit for U^{III} than the larger cryptand. These studies provide insight into the rational design of cryptand-based ligands for trivalent uranium.

Introduction

The accumulation of depleted uranium as a waste product from uranium enrichment encourages the development of research focused on uranium coordination chemistry.[1–3] Reported studies in this area provide insight into potential uses of depleted uranium and fundamental knowledge of uranium coordination chemistry involved in applications such as actinide separations in nuclear waste.[4–8] Within this context, there have been widespread reports of redox-active and redox-inactive ligands used to form complexes of U^{III},[9–16] and one of those ligands, 2.2.2-cryptand, has been widely used to encapsulate metal ions, including U^{III}.[17–19] Further, the coordination chemistry of U^{III}, Np^{III}, and Pu^{III} with 2.2.2-cryptand has been reported recently,[20] expanding cryptand chemistry into the actinides. The thermodynamic and kinetic stability of a cryptate is governed by the cavity size and denticity of the coordinated cryptand as well as the ionic radii and oxidation state of the given metal ion.[21–24] For example, 2.2.1-cryptand fits better with Eu^{III}, and 2.2.2-cryptand fits better with Eu^{II}; moreover, the Gibbs free energy of Eu^{III}(2.2.1-cryptand) is 1.8 times greater than that of Eu^{III}(2.2.2-cryptand), and the dissociation constant of Eu^{III}(2.2.1-cryptand) is 2.7×10^{3} times smaller than that of Eu^{III}(2.2.2-cryptand).[24] Because the size of U^{III} (1.025 Å) is closer to the size of Eu^{III} (0.947 Å) than Eu^{II} (1.17 Å)[25] and the charge density of U^{III} is closer to Eu^{III} than Eu^{II}, we suspected that 2.2.1-cryptand would be a good ligand for U^{III}. Additional support for this suspicion is in our recent report that the flexible counterpart of 2.2.2-cryptand, tris[2-(2-methoxyethoxy)ethyl]amine (TDA-1), forms U^{III}-containing complexes with smaller coordination numbers (nine) compared to all reported 2.2.2-cryptates (with coordination numbers of ten).[26] This report of an acyclic ligand implies that trivalent uranium can be encapsulated by cryptands with smaller denticities than that of 2.2.2-cryptand. Therefore, based on the studies of Eu^{III} cryptand chemistry and U^{III} chemistry with acyclic TDA-1, we hypothesized that 2.2.1-cryptand is a better match for U^{III} than 2.2.2-cryptand. Here, we report U^{III}-containing cryptates of 2.2.2- and 2.2.1-cryptand (Fig. 1) to investigate how ligand denticity affects the structural, spectroscopic, and electrochemical properties of U^{III}.



1 2

Fig. 1 2.2.2-Cryptand, 1, and 2.2.1-cryptand, 2, that were studied with U^{III}. The red color highlights the difference between the two ligands.

[a] Department of Chemistry, Wayne State University, 5101 Cass Avenue, Detroit, MI, 48202, USA. E-mail: mallen@chem.wayne.edu
[b] Lumigen Instrument Center, Wayne State University, 5101 Cass Avenue, Detroit, MI, 48202, USA

† Electronic supplementary information (ESI) available: Crystallographic data, SHAPE analysis, cyclic voltammetry, and electronic absorption spectroscopy. CCDC 2248985–2248987. For ESI and crystallographic data in CIF or other electronic format see DOI: https://doi.org/10.1039/d4dt00521j

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Linn Belford,2013-10-22 Spectroscopy and Photochemistry of Uranyl Compounds is a guide to the research and physics of the actinide elements particularly the uranyl ion The book is introduced with the subject of the spectroscopy of uranyl salts in the solid state Studies dealing with the fluorescence and absorption spectra of solid uranyl salts through band measurements and empirical classification term analysis and fluorescence spectrum in relation to excitation by light of various wavelengths are then discussed The book also mentions the analysis of the uranyl spectrum by Dieke and co workers because of the precise measurements of the fluorescence and absorption bands obtained under the Manhattan project The table determined by Pant and Sakhwalkar in their study of the florescence spectrum of solid hydrated uranyl fluoride at 185 degrees centigrade is presented The text also discusses the theory of electronic structure and spectra of the uranyl ion The spectroscopy of uranyl compounds in solution and the uranyl fluorescence intensity and decay are then presented The book then explains the process of measuring the intensity of slowly decaying florescence of uranyl salts The primary photochemical reactions in uranyl compounds are found to be slow giving rise to many secondary thermal reactions that may be unwanted Researchers in the fields of chemistry and physics working on actinide elements will find this collection of monographs invaluable **Spectroscopic Properties of Inorganic and Organometallic Compounds** ,1987 **Semiannual Report of the Atomic Energy Commission** U.S. Atomic Energy Commission,1956 Radiation Safety and Major Activities in the Atomic Energy Programs U.S. Atomic Energy Commission,1957 **Major Activities in the Atomic Energy Programs** U.S. Atomic Energy Commission,1952 *ERDA Energy Research Abstracts* United States. 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