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

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We describe a study of the influence of cryptand density on the structural, electronic, and electrochemical properties of U<sup>III</sup>-containing cryptates. Two cryptands (2.2.2 and 2.2.1) are reported. The cryptand with the smaller density leads to negative electrochemical potentials and shorter bond lengths that are consistent with a better fit for U<sup>III</sup> 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.<sup>1–3</sup> 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.<sup>4–8</sup> Within this context, there have been widespread reports of redox-active and redox-inactive ligands used to form complexes of U<sup>III</sup>,<sup>9–16</sup> and one of those ligands, 2.2.2-cryptand, has been widely used to encapsulate metal ions, including U<sup>III</sup>.<sup>17–19</sup> Further, the coordination chemistry of U<sup>III</sup>, Np<sup>III</sup>, and Pu<sup>III</sup> with 2.2.2-cryptand has been reported recently,<sup>20</sup> 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.<sup>21–24</sup> For example, 2.2.1-cryptand fits better with Eu<sup>III</sup>, and 2.2.2-cryptand fits better with Eu<sup>II</sup>; moreover, the Gibbs free energy of Eu<sup>III</sup>(2.2.1-cryptand) is 1.8 times greater than that of Eu<sup>III</sup>(2.2.2-cryptand), and the dissociation constant of Eu<sup>III</sup>(2.2.1-cryptand) is 2.7 × 10<sup>5</sup> times smaller than that of Eu<sup>III</sup>(2.2.2-cryptand).<sup>24</sup> Because the size of U<sup>III</sup> (1.025 Å) is closer to the size of Eu<sup>III</sup> (0.947 Å)

than Eu<sup>II</sup> (1.17 Å)<sup>25</sup> and the charge density of U<sup>III</sup> is closer to Eu<sup>III</sup> than Eu<sup>II</sup>, we suspected that 2.2.1-cryptand would be a good ligand for U<sup>III</sup>. 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<sup>III</sup>-containing complexes with smaller coordination numbers (nine) compared to all reported 2.2.2-cryptates (with coordination numbers of ten).<sup>26</sup> 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<sup>III</sup> cryptand chemistry and U<sup>III</sup> chemistry with acyclic TDA-1, we hypothesized that 2.2.1-cryptand is a better match for U<sup>III</sup> than 2.2.2-cryptand. Here, we report U<sup>III</sup>-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<sup>III</sup>.

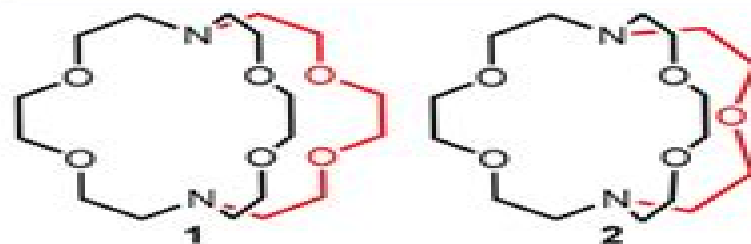


Fig. 1 2.2.2-Cryptand, 1, and 2.2.1-cryptand, 2, that were studied with U<sup>III</sup>. The red color highlights the difference between the two ligands.

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†Electronic supplementary information (ESI) available: Crystallographic data, SHAPE analysis, cyclic voltammetry, and electronic absorption spectroscopy. CCDC 2244995–2244997. For ESI and crystallographic data in CIF or other electronic format see DOI: <https://doi.org/10.1039/D4DT00052J>



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