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Uranium amides as precursors to cationic and/or pentavalent compounds

M. Ephritikhine*, J.C. Berthet, C. Boisson, M. Lance, M. Nierlich

Service de Chimie Moléculaire, DSM, DRECAM, CNRS URA 331, CEA Saclay, 91191 Gif sur Yvette, France

Abstract

The uranium chloroamide complexes $[U(NEt_2)_nCl_{4-n}]$ ($n=1$, 2) were isolated from the comproportionation reactions of $[U(NEt_2)_4]$ and UCl_4. The protonolysis reaction of the $M–NR_2$ bond (M=Ti, Zr, Hf, U; R=Me, Et, $SiMe_3$) with the ammonium salt NHR'_3BPh_4 (R′=Et, Me) constitutes an efficient and practical route to cationic complexes. Thus were prepared a series of organometallic uranium cations in the +3, +4 and +5 oxidation states. The reactions of these cationic compounds with nucleophiles, proton acidic substrates and unsaturated molecules have been developed. The dialkylamide ligand was useful to stabilize unique examples of neutral and cationic complexes of uranium in the +5 oxidation state. © 1998 Elsevier Science S.A.

Keywords: Uranium; Amides; Cations; Pentavalent

1. Introduction

In 1956, Gilman et al. isolated the first uranium amide compound, $[U(NEt_2)_4]$ [1], but it is only after a period of stagnation that the chemistry of the uranium amide complexes has been considered and since the 1970s, it has been developed regularly [2]. The amide ligands in these complexes are essentially the NEt_2 group, which is found only in compounds of uranium(IV), and the $N(SiMe_3)_2$ group which is able to stabilize all the oxidation states of the metal, from +3 to +6. Not surprisingly, the structure of the complexes is strongly influenced by the size of the amide group. In the solid state, the tetramides $[U(NR_2)_4]$ adopt a trimeric, a dimeric and a monomeric structure for R=Me [3], Et [4] and Ph [5], respectively.

However, the main interest in these uranium amide complexes is their potential in inorganic and organometallic synthesis. Two reactions have been largely used for the preparation of new complexes: the substitution of the amide group with proton acidic substrates (Eq. (1)) and the insertion of unsaturated dipolar molecules into the metal-nitrogen bond (Eq. (2)).

$$[\{U\} - NR_2] + HA \rightarrow [\{U\} - A] + HNR_2 \qquad (1)$$

$$[\{U\} - NR_2] + A{=}B \rightarrow [\{U\} - A - B - NR_2] \qquad (2)$$

Here we present our contribution to the development of the chemistry of uranium amide complexes. After the synthesis of new chloro-amide compounds, we found a novel protonolysis reaction of the amide ligand which constitutes an efficient method of preparation of cationic complexes. These exhibited an interesting reactivity and proved to be valuable precursors of a series of organometallic derivatives. We also found that the dialkylamide group was quite useful for the stabilization of unique uranium complexes in the +5 oxidation state.

2. The chloro-amide complexes

We envisaged the preparation of some new complexes by using the metathesis reaction (Eq. (3)) and we wished to have at our disposal the chloroamide precursors;

$$[Cl - \{U\} - NR_2] + MA \rightarrow [A - \{U\} - NR_2] + MCl \qquad (3)$$

such complexes of the f elements are rare, being limited to the disilylamide derivatives $[M\{N(SiMe_3)_2\}_nCl_{4-n}]$ ($n=3$ and M=Th or U [6]; $n=2$ and M=U [7]). The most straightforward route to $[U(NEt_2)Cl_3(THF)]$ and $[U(NEt_2)_2Cl_2]$ is the comproportionation reaction between $[U(NEt_2)_4]$ and UCl_4, giving the products in very good yields [8]. The tris diethylamide derivative $[U(NEt_2)_3Cl]$ was found to be in equilibrium with $[U(NEt_2)_4]$ and $[U(NEt_2)_2Cl_2]$. These chloroamide complexes form stable adducts with Lewis bases, like $[U(NMe_2)_3Cl(HMPA)_2]$ (HMPA=hexamethylphosphoramide) which exhibits a *cis* octahedral structure in the solid state [3]. It is interesting to

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*Corresponding author. Fax: +33 1 69086640; e-mail: ephri@nanga.saclay.cea.fr

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note that $[U(NEt_2)_4(HMPA)_2]$ also exists in solution in the preferred *cis* configuration, in contrast to the other compounds $[UX_4(HMPA)_2]$ (X = Cl, Br [9], BH_4 [10], SiPr [11]) which invariably adopt a *trans* configuration. These distinct structural features indicate that the dialkylamide ligand would have a strong *trans* influence.

The chloroamide complexes can serve as precursors for the synthesis of new compounds. For example, treatment of $[U(NEt_2)_2Cl_2]$ with the potassium salt of the cyclooctatetraene dianion afforded $[U(cot)(NEt_2)_2(THF)]$ (cot = η-C_8H_8) in almost quantitative yield [12]. This reaction represents a convenient entry into the chemistry of the monocyclooctatetraene uranium compounds, which has been developed in recent years [13–18]. However, it appeared that such metathesis reactions could be impeded by the easy formation of anionic complexes, with retention of the alkali metal in the product; moreover, elimination of lithium or sodium chloride from the reaction mixture is not always straightforward. In order to circumvent these difficulties, which are quite common in f element chemistry, we have devised a new route, represented by Eq. (4), i.e the addition of A^- to a cationic amide species

$$[\{U\} - NR_2][BPh_4] + MA \rightarrow [A - \{U\} - NR_2] + MBPh_4 \tag{4}$$

3. The cationic complexes

3.1. Synthesis

Cationic complexes are most generally prepared by abstraction of halide or alkyl ligands with silver reagents or by protonation of metal-hydrogen or metal-carbon bonds with ammonium salts. Our attempts to abstract chloride ions from the chloroamide complexes have been unsuccessful. By analogy with the protonation of alkyl compounds, we considered the protonolysis of a metal-amide bond by means of $NHEt_3BPh_4$ and we found that this reaction constitutes an efficient synthesis of cationic complexes. While the reactivity of the metal-amide bond towards acidic proton substrates was well established, the protonolysis reaction (Eq. (5)) was unprecedented. This reaction is rapid, giving the products in very good yields; the liberated amines are easily eliminated from the reaction mixture.

$$[\{U\} - NEt_2] + NHEt_3BPh_4 \rightarrow [\{U\}][BPh_4] + NHEt_2 + NEt_3 \tag{5}$$

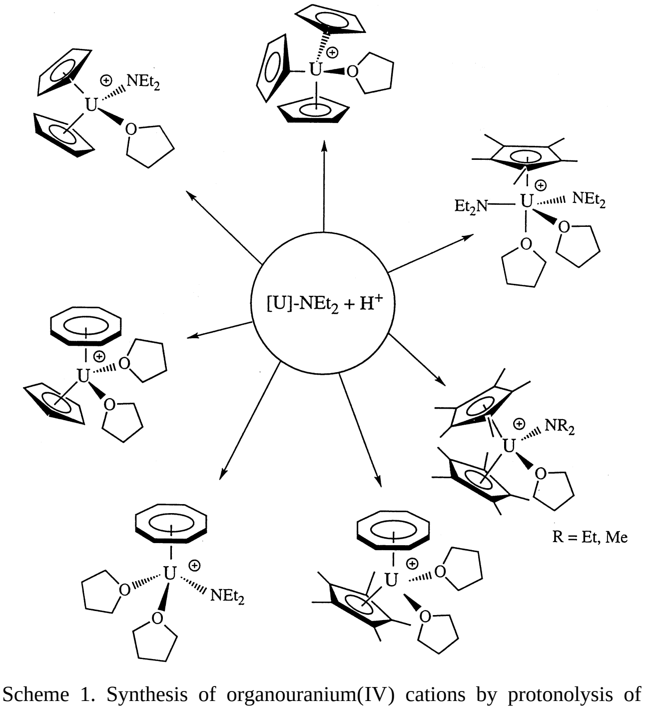
The cationic complexes $[U(NEt_2)_3][BPh_4]$, $[U(NEt_2)Cl_2(THF)_2][BPh_4]$ and $[UCl_3(THF)_2][BPh_4]$ were thus isolated from the reactions of the corresponding neutral precursors with one equivalent of $NHEt_3BPh_4$ in THF [8]. The dicationic complexes $[U(NEt_2)_2(THF)_3][BPh_4]_2$ and $[UCl_2(THF)_4][BPh_4]_2$ were synthesized by further protonation of the above cationic amide compounds in refluxing THF. The crystal structures of $[U(NEt_2)_3(THF)_3][BPh_4]$ and $[U(NEt_2)_2(NC_5H_5)_5][BPh_4]_2$ indicated that the uranium-nitrogen bond distances are quite similar and average 2.18 Å [8].

Protonation of the $N(SiMe_3)_2$ ligand was much more difficult than that of the diethyl or dimethylamide group. As an illustration, treatment of the mixed amide compound $[U(NEt_2)_2(N\{SiMe_3\}_2)_2]$ with $NHEt_3BPh_4$ readily gave the cation $[U(NEt_2)(N\{SiMe_3\}_2)_2]^+$ in quantitative yield [19]. This remarkable selectivity can be easily explained by the steric and electronic effects of the trimethylsilyl group which impede the attack of the acidic reagent onto the nitrogen atom of the amide ligand.

For testing the potential of the protonolysis reaction (Eq. (5)) in the synthesis of cationic complexes of the d transition metals, we considered its application to some representative amide derivatives of the Group 4 elements [20]. The cations $[Ti(NMe_2)_3(THF)]^+$ and $[M(NMe_2)_3(THF)_2]^+$ (M = Zr, Hf) were thus easily obtained in very good yields; these are unique examples of tris(dialkylamide) derivatives of these metals. The trigonal bipyramidal crystal structure of the cation $[Ti(NMe_2)_3(NC_5H_5)_2]^+$ has been determined.

The protonolysis reaction (Eq. (5)) served to prepare a variety of cationic organometallic compounds. In Scheme 1 are represented the series of uranium(IV) derivatives which have been obtained from the corresponding neutral dialkylamide precursors [21].

These are the first examples of cationic monocyclopen-



Scheme 1. Synthesis of organouranium(IV) cations by protonolysis of amide precursors.

tadienyl, biscyclopentadienyl, cyclooctatetraene and mixed ring complexes of uranium(IV). The convenient synthesis of these complexes emphasize the general applicability of the protonolysis reaction (Eq. (5)). The latter is much more efficient than protonolysis of the alkyl analogues, which are less reactive or inert towards the ammonium salt, and show the advantage of the amide compounds as precursors to cationic species. The crystal structures of $[U(cp^*)(NEt_2)_2(THF)_2][BPh_4]$ [21], $[U(cot)(NEt_2)(THF)_3][BPh_4]$ [22], $[U(cp^*)(cot)(THF)_2][BPh_4]$ [21] and $[U(cp^*)_2(NMe_2)(CNtBu)_2][BPh_4]$ [23] have been determined; they exhibit similar metal-nitrogen distances, typically equal to 2.18–2.22 Å, which are at the lower limit of the range of uranium-nitrogen bond distances for terminally coordinated amide groups.

The protonolysis reaction (Eq. (5)) also served to prepare the first cationic metallocene compound of uranium(III), $[U(cp^*)_2(THF)_2][BPh_4]$ [19]. The $N(SiMe_3)_2$ group being inert towards $NHEt_3BPh_4$, protonation of $[U(cp^*)_2(N\{SiMe_3\}_2)]$ was achieved by using NH_4BPh_4 (Eq. (6)); the greater reactivity of the latter can be accounted for by its smaller size and its more important acidic character. In the crystal, the U(III) cation is isostructural with its samarium analogue [24].

$$[U(cp^*)_2(N\{SiMe_3\}_2)] + NH_4BPh_4 \rightarrow [U(cp^*)_2(THF)_2][BPh_4] + NH_3 + NH(SiMe_3)_2 \qquad (6)$$

The first uranium(V) cation, $[U(cot)(NEt_2)_2(THF)]^+$, was synthesized by using reaction (5) [12] (vide infra).

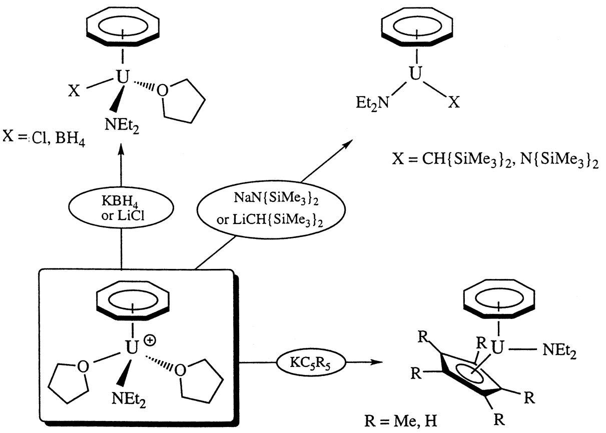
3.2. Reactions of the cationic amide complexes

Cationic amide complexes of the d transition metals and f elements are very rare, being limited to a few monoalkylamido derivatives [25–27]; such compounds are however expected to exhibit a great reactivity. We studied in more detail the reactions of $[U(cot)(NEt_2)(THF)_2][BPh_4]$ [22] and $[U(cp^*)_2(NMe_2)(THF)][BPh_4]$ [23].

3.2.1. Addition reactions

A series of neutral compounds have been obtained by treatment of the monocyclooctatetraene cation with the alkali metal salts of various anionic reagents (Scheme 2). These compounds were isolated in good yields, except the chloride and borohydride derivatives which decomposed by desolvation [22].

These results underline the superiority of these addition reactions over the classical substitution reactions of halide complexes. Moreover, it is noteworthy that the cations $[U(cot)(NEt_2)(THF)_2]^+$ and $[U(NEt_2)_3]^+$ are stable, in contrast to the corresponding chlorides $[U(cot)(NEt_2)Cl]$ and $[U(NEt_2)_3Cl]$ and are therefore of special interest as starting materials.

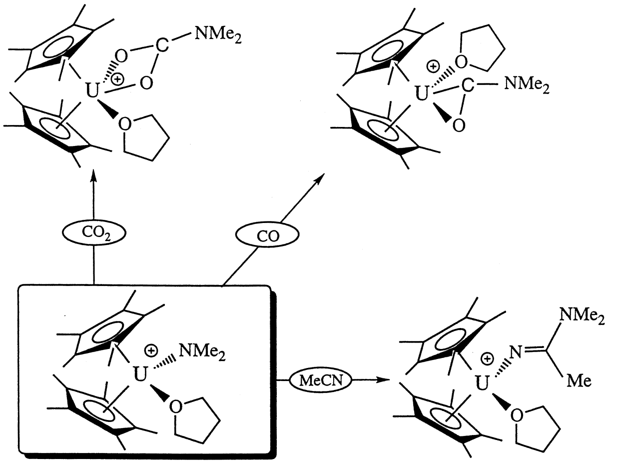


Scheme 2. Addition of nucleophiles to the cation $[U(cot)(NEt_2)(THF)_2]^+$.

3.2.2. Insertion and substitution reactions

Insertion of unsaturated molecules into the metal-nitrogen bond and substitution of the amide ligand with proton acidic substrates are interesting since they lead to the formation of new cationic complexes. Cations are very rare in organouranium chemistry and in the family of the bispentamethylcyclopentadienyl compounds, which was of fundamental importance in the development of this discipline, the only cations are those here reported: $[U(cp^*)_2(THF)_2]^+$ [19], $[U(cp^*)_2(NMe_2)(THF)]^+$ [23] and $[U(cp^*)_2(NEt_2)_2]^+$ (vide infra) which are, respectively, in the +3, +4 and +5 oxidation states. The U(IV) cation was readily and cleanly transformed into the corresponding cationic amidino and carbamate derivatives, by insertion of acetonitrile and carbon dioxide into the uranium-nitrogen bond (Scheme 3). Surprisingly, the amide cation reacted rapidly with carbon monoxide in THF to give the carbamoyl compound which, from the spectroscopic data, adopts a dihapto configuration.

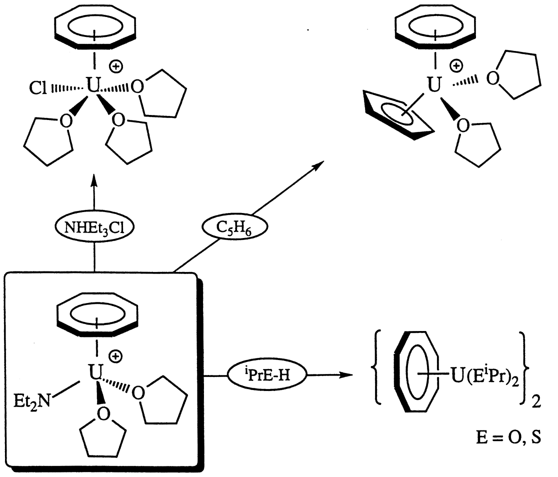
Similar reactions were observed with the monocyclooctatetraene cation $[U(cot)(NEt_2)(THF)_2]^+$ [22]. Its treatment with carbon disulfide afforded



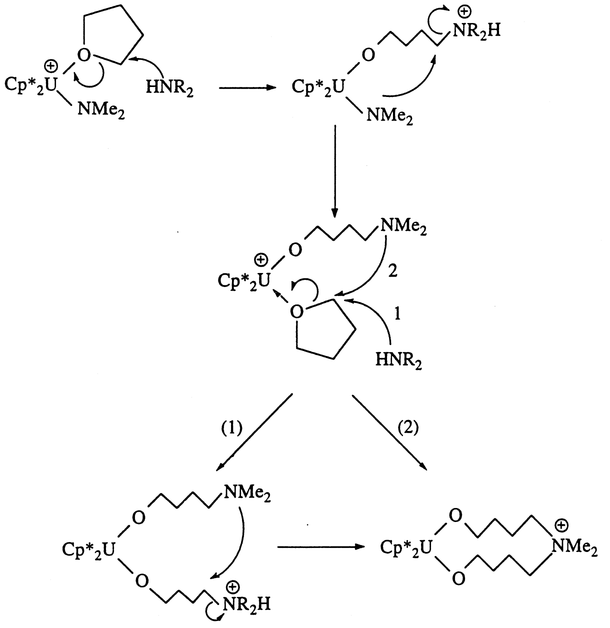
Scheme 3. Insertion reactions of the cation $[U(cp^*)_2(NMe_2)(THF)]^+$.

$[U(cot)(S_2CNEt_2)(THF)_2][BPh_4]$, the first uranium(IV) dithiocarbamate to have been crystallographically characterized.

The cationic amide complexes and their neutral amide precursors exhibit major differences in their reactivity. Thus, in contrast to the cation $[U(cot)(NEt_2)(THF)_2]^+$ which undergoes the insertion of acetonitrile into the metal-nitrogen bond, the corresponding neutral bis amide complex $[U(cot)(NEt_2)_2(THF)]$ reacts with this molecule to give the substitution product, with liberation of amine. These results revealed that the cationic amide complex was less sensitive to acids than the corresponding neutral bis amide compound. This feature was further illustrated with reactions with other acidic substrates [22] (Scheme 4). Formation of the cation $[U(cot)(cp)(THF)_2]^+$ was much slower than that of $[U(cot)(cp)(NEt_2)]$ from $[U(cot)(NEt_2)_2(THF)]$. The cation $[U(cot)(NEt_2)(THF)_2]^+$ was inert towards the less acidic phenylacetylene, in contrast to the neutral bis amide which was readily transformed into the acetylide $[U(cot)(NEt_2)(C{\equiv}CPh)]$. The distinct reactivity of the monocyclooctatetraene cation towards isopropanol and phenylacetylene, which have similar acidities, can be accounted for by metal coordination of the alcohol to the cation, giving the adduct $[U(cot)(NEt_2)(iPrOH)]^+$, followed by proton transfer and elimination of free amine. The intermediate $[U(cot)(OiPr)(THF)_x]^+$, which was observed by NMR, would itself coordinate an alcohol molecule and $[U(cot)(OiPr)(iPrOH)]^+$ would be deprotonated into $[U(cot)(OiPr)_2]$ by $NHEt_2$. Like the monocyclooctatetraene cation, $[U(cp^*)_2(NMe_2)(THF)]^+$ was rather inert towards proton acidic molecules unless these could be activated by prior coordination to the metal centre [23]. The monocyclopentadienyl complex $[U(cp^*)(NMe_2)_2(THF)_2][BPh_4]$ reacted with two equivalents of ethanol to give $[U(cp^*)(OEt)_2(THF)_2][BPh_4]$ in 82% yield, according to Eq. (7) [23].



Scheme 4. Reactions of the cation $[U(cot)(NEt_2)(THF)_2]^+$ with proton acidic substrates.



Scheme 5. Proposed mechanism for the ring opening reaction of THF promoted by the cation $[U(cp^*)_2(NMe_2)(THF)]^+$.

$$[U(cp^*)(NMe_2)_2(THF)_2][BPh_4] + 2EtOH \rightarrow [U(cp^*)(OEt)_2(THF)_2][BPh_4] + 2NHMe_2 \qquad (7)$$

3.2.3. Ring opening reaction of THF promoted by $[U(cp^*)_2(NMe_2)(THF)][BPh_4]$

Another characteristic of cationic complexes is their Lewis acidity which is often evidenced by the ability to promote the ring opening reaction of THF. The complex $[U(cp^*)_2(NMe_2)(THF)][BPh_4]$ was readily prepared in this solvent and was isolated in very good yield. However, by keeping the reaction mixture for a longer period at room temperature, this compound was slowly transformed into the metallacycle $[(cp^*)_2U(O(CH_2)_4NMe_2(CH_2)_4O)][BPh_4]$ which was characterized by its crystal structure [23,28]. This complex is a unique example of a heterocyclic metallacycle containing both oxygen and nitrogen atoms. A mechanism for its formation is proposed in Scheme 5; the free amine plays a catalytic role in the reaction.

While nucleophilic ring opening reactions of THF are well documented, this synthesis provides the first example of such a transformation where two molecules are opened on a single metal centre to give a heterocyclic metallacycle.

4. The uranium(V) complexes

The dialkylamide group may be regarded as a relatively hard ligand, although not to the extent of the alkoxide and fluoride groups, and is therefore able to stabilize the high oxidation states of the metals. When we started this work, the only organometallic complexes in the +5 oxidation state were a few imido derivatives which were synthesized by oxidation of trivalent cyclopentadienyl precursors with the corresponding organic azide [29–32]. Our initial strategy for obtaining uranium (V) amide compounds was the oxidation of anionic tetravalent precursors.

The pentaamide anion $[U(NEt_2)_5]^-$ was isolated in good yield after treatment of UCl_4 with $LiNEt_2$ or addition of the latter to $[U(NEt_2)_4]$ [33]. Similar addition of $LiNMe_2$ to $[U(NMe_2)_4]$ afforded the dianion $[U(NMe_2)_6]^{2-}$ [3]. Oxidation of these two compounds by means of $TlBPh_4$ readily gave the neutral pentaamide $[U(NEt_2)_5]$ and the anionic hexaamide $Li[U(NMe_2)_6]$, according to Eq. (8) and Eq. (9).

$$Li[U(NEt_2)_5] + TlBPh_4 \rightarrow [U(NEt_2)_5] + Tl + LiBPh_4 \quad (8)$$

$$Li_2[U(NMe_2)_6] + TlBPh_4 \rightarrow Li[U(NMe_2)_6] + Tl + LiBPh_4 \quad (9)$$

Further oxidation of the monoanion gave $[U(NMe_2)_6]$, the unique example of a uranium(VI) amide complex; the latter was moderately stable in solution and, at this time, is characterized only by its NMR spectrum [4]. Addition of $LiNEt_2$ to $[U(cot)(NEt_2)_2(THF)]$ but also to $[U(cot)_2]$ gave the anion $[U(cot)(NEt_2)_3]^-$ which was alternatively prepared by treating $[U(NEt_2)_4]$ or $[U(NEt_2)_3][BPh_4]$ with K_2cot [12]. Oxidation of this anion with thallium or silver tetraphenylborate readily afforded the corresponding neutral uranium(V) compound $[U(cot)(NEt_2)_3]$ in quantitative yield (Eq. (10)).

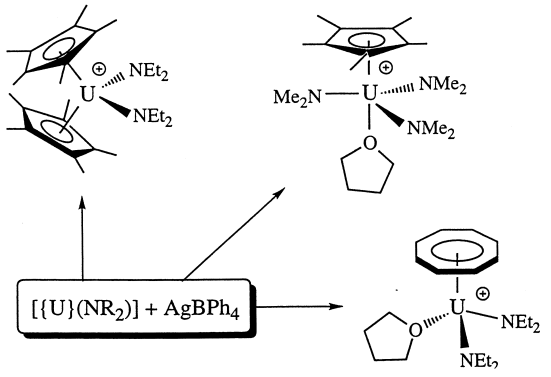
$$Li[U(cot)(NEt_2)_3] + TlBPh_4 \rightarrow [U(cot)(NEt_2)_3] + Tl + LiBPh_4 \quad (10)$$

By alcoholysis with 2-propanol, this latter was transformed into $[U(cot)(OiPr)_3]$ which was alternatively prepared by oxidation of the anion $[U(cot)(OiPr)_3]^-$. The neutral complex $[U(cot)(NEt_2)_2(THF)]$ could not be oxidized with $TlBPh_4$ but its treatment with thallium cyclopentadienyl readily afforded the mixed ring uranium(V) compound $[U(cot)(cp)(NEt_2)_2]$ with precipitation of metallic thallium (Eq. (11)).

$$[U(cot)(NEt_2)_2(THF)] + Tlcp \rightarrow [U(cot)(cp)(NEt_2)_2] + Tl \quad (11)$$

It is likely that this reaction proceeds by initial addition of the cyclopentadienyl ligand, giving the anion $[U(cot)(cp)(NEt_2)_2]^-$, followed by oxidation with thallium(I). In agreement with this mechanism, the mixed ring anion was prepared and oxidized into $[U(cot)(cp)(NEt_2)_2]$ with $TlBPh_4$.

In the presence of the more oxidizing silver salt $AgBPh_4$, $[U(cot)(NEt_2)_2(THF)]$ was transformed into $[U(cot)(NEt_2)_2(THF)][BPh_4]$. This cation was also obtained by protonolysis of $[U(cot)(NEt_2)_3]$ with $NHEt_3BPh_4$ [12]. Other neutral organouranium(IV) amide complexes could be oxidized in a similar way (Scheme 6).



Scheme 6. Synthesis of organouranium(V) cations.

Thus, $[U(cp^*)(NMe_2)_2(THF)][BPh_4]$ and $[U(cp^*)_2(NEt_2)_2][BPh_4]$ were prepared in good yield from their corresponding neutral precursors [34]. It is noteworthy that these oxidation reactions represent a new synthetic route to organouranium cations. These complexes, which are the first cationic derivatives of uranium(V), have been crystallographically characterized.

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