Dinuclear uranium complexation and manipulation using robust tetrararylides†

Jordann A. L. Wells, Megan L. Seymour, Markéta Suvova and Polly L. Arnold*

Two lower-oxidation state uranium cations can be readily combined in a robust, yet flexible and derivatisable, tetrararylide ligand framework, affording a new platform at which to use the multi-electron reductive capacity of the two actinide centres.

While single organometallic uranium centres are often capable of binding and reducing small molecules such as dinitrogen and carbon oxides, the most notable levels of activation and transformations are achieved almost exclusively by the combination of two uranium cations around one substrate.1,2

For example, dinitrogen overpressures as high as 80 psi are required to stabilise the terminal [(Cp*)2U(η1−N2)],3 and the first molecular uranium carbonyl [(Me3SiC5H4)3UCO] showed reversible CO binding in solution.5 However, two molecules of uranium tris(aryl oxide) or tris(siloxide) UX3 (X = O-2,4,6-′Bu3C6H2, OSi(2,4,6-′Me4C6H2)) in combination can effect the reduction of N2 to such an extent that the molecule [X3UIV[μ-η3:η3-N2]] is stable in boiling toluene, and can reductively couple CO at ambient temperature and pressure to the ynediolate [X3UHIV(μ-OOC)] with further C–H and C–C bond formations possible. The conversion of allyl C–H to C–B bonds has also been shown to be possible in di-uranium(arene) complexes [X2UIV(μ-η6–η5–η7–C6H5R)UHVX2] (R = H, alkyl, aryl).7 The recently reported reductive activation of CO by pairs of the uranium complexes [U[η(–C6H6SiR3)2][η–Cp][η–Cp]] (R = Me,1 Pr′; R′ = Me4H, Me3, Me3SiMe3, Me3Et) has been particularly instructive in showing that bonds between the two uranium centres depend on the steric accessibility to the two U centres (rather than the redox capability).8,9

All these results suggest that a ligand pre-organised to hold two reducing U centres would be desirable if these small molecule activations are to be rendered catalytic, or better controlled. In collaboration with Love, we recently reported the use of expanded Pacman-shaped N-donor macrocycles to combine two UIII centres at a distance suitable for trapping a di- or triatomic fragment, but have been unable as yet to isolate complexes in which no X-ligand is coordinated between the two U centres.10,11

Recognising the strength of the U–aryl oxide bond in a range of U oxidation states,7,12–16 we have developed a two-hour, one-pot, large-scale synthesis of three closely related analogues of a known arene-bridged tetraphenol17 in order to isolate and study the first O-donor compounds containing two discrete UIII or UIV centres in a single molecule, in geometries pre-organised for small molecule binding. The three phenols used here are H4Lp and H4LM, and phenyl-substituted H4LP±, Fig. 1.

Bimetallic salts of the phenols closely related to H4LP and H4LM in Fig. 1 (with R1 = R2 = ‘Bu) have been demonstrated to be excellent ring opening polymerisation initiators for monomers including lactide (by H2K2LP and H2K2LM adducts),18 epoxide (by bis-AlIII adducts of LP with R1 = R2 = ‘Bu),19 and ε-caprolactone (by bis-Nb or Ta adducts of LP with R1 = R2 = ‘Bu).20 X-ray structural analyses in some of these complexes demonstrate a ligand flexibility that enables the metals to reside on the same or opposite sides of the central arene bridge.19,21

![Fig. 1](image-url)
Both salt metathesis and protonolysis routes allow access to diuranium complexes of the tetraphenolates, as shown in Scheme 1.

First, treatment of the \textit{in situ} formed dicalcium salt Ca$_2$L$_R^+$ (R = P, P*) or tetrapotassium K$_4$L$_R^+$ (R = M) with two equivalents of \textit{UI}$_4$(diox)$_2$ (diox = O(CH$_2$CH$_2$)$_2$O, 1,4-dioxane) in THF or dioxane affords the green crystalline diuranium target complexes after work-up to remove salt by-products. The di-volatile, hexane soluble by-product HN$_2$ can be isolated after work-up in excellent yields (65–80%).

Second, treatment of the proligand H$_4$L$_R$ with two equivalents of the uranium(IV) complexes \textit{UIV}$_n$S$_2$P$_*$(SiMe$_3$)$_2$ results in full deprotonation of all four acidic phenols to afford the unsolvated, yellow, crystalline \textit{UN}_n$S_2$P$_*$$_2$(SiMe$_3$)$_2$ complexes after work-up to eliminate the volatile, hexane soluble by-product HN$_2^*$, in essentially quantitative yields.

Complexes \textit{1R} and \textit{2R} have been fully characterised, including by single crystal X-ray diffraction. The \textit{in-situ} synthesis of the calcium salts are described below as they have proven ideal metathesis precursors for some, since Group 1 bases often afford compounds that retain one or more bridging aryloxide protons.$^{18}$

In our hands, direct syntheses of uranium(III) analogues of \textit{1R} and \textit{2R} from uranium(IV) halide and amide starting materials were unsuccessful. We therefore investigated the electrochemical and chemical reduction of the uranium(IV) complexes. The $^{1}$UIV redox couple is known to range from $-2.78$ to $-1.83$ V \textit{versus} ferrocene depending on the ligand environment.$^{1,22,23}$ The cyclic voltammetry data show that the complexes \textit{1R} and \textit{2R} have one wave in the negative potential region attributable to the single electron reduction of both metal centres at the same time, and confirming the absence of $^{1}$UV$^{-}$–$^{1}$UIV electronic communication through the ligand in all cases. The potentials of the complexes are collated in Table 1 and suggest that the uranium(III) complexes should be chemically accessible from a reaction with common one-electron reductants such as group 1 metals. The treatment of \textit{2M} with two equivalents of KC$_8$ affords dark purple di-$^{1}$UIIII \textit{UN}_n$S_2$P$_*$$_2$LM, \textit{3M} in 63% yield after workup, which has been characterised by multinuclear NMR spectroscopy and elemental analysis, Scheme 1.

Complexes \textit{1}, \textit{2} and \textit{3} are soluble in hot THF, dioxane, pyridine and arene solvents. The $^1$H NMR spectra of the iodide complexes \textit{1} are moderately shifted by the \textit{UIV} centres with resonances in the range 14 to 0 ppm whereas the amide complexes \textit{2} are more significantly shifted with proton resonances spanning from 40 to $-20$ ppm.

Interestingly, following reduction of \textit{2M} to give \textit{3M}, the chemical shift range is decreased and proton resonances occur between 22 and $-13$ ppm. The $^{29}$Si resonance of the silylamide atom occurs at around $-230$ ppm for both \textit{2P} and \textit{2P*} and is shifted to $-100$ ppm in \textit{3M}.

Single crystals of \textit{1} and \textit{2} were grown, details for which are in the ESI.$^\dagger$ The molecular structures of \textit{1P}, \textit{1P*}, \textit{1M}, \textit{2P} and \textit{2P*} are shown in Fig. 2 and 3; that for \textit{2P*} is in the ESI$^\dagger$ along with the structure of a dioxane solvate of \textit{1P}, \textit{1P}(dioxane).

The uranium centre in \textit{1P} is seven-coordinate, adopting square face monocapped trigonal prismatic geometry, whereas the six coordinate uranium centres in \textit{1P*} and \textit{1M} adopt distorted octahedral geometry. The dioxane adduct of \textit{1P}, \textit{1P}(dioxane), also displays six coordinate uranium centres in distorted octahedral geometry. The equatorial plane is occupied by the aryloxide and iodide ligands, and the axial positions occupied by coordinated dioxane molecules. The exo-

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Table 1. Selected reduction potentials versus $\text{Fc}^-$/\text{Fc} measured in THF using 0.1 M $[^{13}$Bu$_4$N][BPH$_4$] as the supporting electrolyte.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Reduction potential at 100 mV s$^{-1}$/V</th>
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<tbody>
<tr>
<td>$^{1}$M</td>
<td>$-2.03$</td>
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<tr>
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<td>$-2.05$</td>
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<tr>
<td>$^{2}$P*</td>
<td>$-1.53$</td>
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axial dioxanes act as a bridging ligand, linking the uranium centres in separate molecules to form a one-dimensional polymer in the solid state (see ESI† for further information).

The coordination environment of the two uranium centres in 1M differs. While both metal centres have a pseudo-octahedral geometry, the aryloxide and iodide ligands occupy the equatorial plane about U2 with the axial positions occupied by THF donor molecules in a trans arrangement. The THF donors about the U1 centre, however, are mutually cis occupying one equatorial and one axial position. The two iodides and one aryloxide group occupy the three remaining equatorial positions and the other aryloxide occupies the axial position. This surprising feature results in unsymmetrical bond lengths and angles in the solid state. The U1–O2 bond length is slightly shorter than the average of the three other bond lengths (2.080(11) Å and 2.124(10) Å respectively). Perhaps most notable is the distortion of the U1–O1–Cipso angle of 138.7(9)° compared to the average of the other three angles, 157.0(9)°.

The U–OAr bond distances in 1P, 1P* and 1M are very similar, with average distances of 2.112(5) Å, 2.106(4) Å and 2.120(10) Å respectively. These are comparable to previously reported uranium(iv) bis(aryloxo) bis(iodo) complexes such as I₂U(ODtbp)₂(THF) (ODtbp = O–2,6-iBu₂C₆H₄) with an average U–O bond length of 2.076 Å,¹⁴ and I₂U(ArO)₂(THF)₃ (Ar = O–4’-BuC₆H₄, O–2,6-Me₂C₆H₄, C₆F₅) with average U–O distances of 2.068(8) Å, 2.091(8) Å and 2.120(6) Å respectively.²⁴
The U–O–C$_{ipso}$ bond angles for the 1$^R$ complexes are bent, reminiscent of the homoleptic uranium(IV) complex U(ODtbp)$_4$, with angles of 157.1(4)$^\circ$, 156.0(3)$^\circ$, 152.4(9)$^\circ$ for 1$^P$, 1$^P*$ and 1$^M$ respectively, compared to 154.04(8)$^\circ$ for U(ODtbp)$_4$. This is somewhat unusual, as the U–O–C$_{ipso}$ bond angles of other complexes of the type I$_2$U(OAr)$_2$ fall within the range 166.2(8)$^\circ$ to 176.9(8)$^\circ$, and could be ascribed to the constraints imposed by the ligand frame.

The four-coordinate uranium centres in complexes 2$^P$, 2$^P*$ and 2$^M$ all adopt a distorted tetrahedral geometry. As shown in Table 2, the complexes have comparable average U–O bond distances of 2.1198(13) Å, 2.1422(19) Å and 2.122(4) Å respectively, which is also true of the U–N bond distances of 2.2530 (16) Å, 2.257(2) Å and 2.252(6) Å respectively. The slight elongation of U–O and U–N bonds in 2$^P*$ compared to 2$^P$ and 2$^M$ can be rationalised by the increased steric bulk around the metal centre in the larger tetraaryloxide framework interacting with the sterically demanding silylamine ligands. Similarly to the iodide complexes, the U–O–C$_{ipso}$ bond angles are closer to the homoleptic uranium(IV) aryloxide, as opposed to the I$_2$U(OAr)$_2$ analogues, with mean angles of 149.47(12)$^\circ$, 150.72(17)$^\circ$ and 148.5(4)$^\circ$ for 2$^P$, 2$^P*$ and 2$^M$ respectively. The U–O bond distances are comparable to that of U(ODtbp)$_4$, as well as the tetrahedral mixed aryloxo-amido uranium(IV) complexes Et$_3$NU(ODtbp)$_3$ and N$_3$U(ODtbp) with average U–O distances of 2.143(4) Å and 2.145(8) Å respectively. The U–N bond distances of 2$^R$ differ from the U–N distance of 2.161(5) Å exhibited by Et$_3$NU(ODtbp)$_3$, slightly, but agree very well with the U–N bond distances exhibited by N$_3$U(ODtbp) and the uranium (v) complex N$_3$U(Onaph)$_2$ (napth = C$_{10}$H$_7$) of 2.284(10) Å and 2.222(6) Å respectively. This discrepancy in the U–N bond distance is presumably due to the difference in steric environment imposed by the bis(trimethylsilyl)amide ligand compared with that of the diethyldiamine ligand.

Inspection of the solid-state structures of these binmetallic derivatives, and of literature examples of other metal complexes suggests that the coordination of the two metals on the same side of the arene bridge is the preferred geometry in the meta-substituted L$^M$ complexes, but significantly rarer in the others, but is presumably not retained in solution for the smaller R substituted phenols. This could be due to the additional stability afforded by the generation of a dipole across the molecule.

### Table 2: Comparison of key metrics for the di-uranium complexes

<table>
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<tr>
<th></th>
<th>1p</th>
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<th>2*</th>
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<tr>
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</tbody>
</table>

### Conclusions

Straightforward syntheses of dinuclear U$^{IV}$ complexes are possible using three different tetraphenolate ligands, in which a para- or meta-substituted phenyl backbone provide a strong yet flexible support to the two metal centres. The dinuclear U$^{III}$ analogues are most readily accessible by chemical reduction. The meta-arene bridged ligand appears to favour the coordination of both metals on the same side of the bridge, a factor which may enhance the ability to reductively couple certain small molecules.

**Meta**- and *para*-functionalised aryl imido and alkynide ligands have previously been used to demonstrate viable magnetic exchange between $f^1$ uranium centres. The properties are switched by changing the substitution patterns of the linking arene groups, and have been suggested to be of use in developing f-block magnetic materials for data storage, quantum computing or refrigeration applications. In addition to reactivity studies of these new potential multi-electron reductants, work is in progress to understand the magnetic behaviour of these new complexes.

### General details

All moisture and air sensitive materials were manipulated using standard high-vacuum Schlenk-line techniques and MBraun gloveboxes and stored under an atmosphere of dried and deoxygennated dinitrogen. All gases were supplied by BOC gases UK. All glassware items, cannulae and Fisherbrand 1.2 µm retention glass microfibre filters were dried in a 160 °C oven overnight before use.

Tetrahydrofuran and hexane for use with moisture and air sensitive compounds were dried using a Vac Atmospheres solvent purification system and stored over activated 4 Å molecular sieves. The solvent was cycled through a drying column containing molecular sieves for 12 hours before collection. 1,4-Dioxane for use with moisture and air sensitive compounds was refluxed over sodium for 3 days, distilled and collected into an ampoule containing 4 Å molecular sieves. All solvents were degassed and stored for 2 days prior to use. d$_6$-Benzene and d$_4$-tetrahydrofuran were freeze pump thaw degassed, refluxed over potassium for 24 hours and distilled by trap to
trap distillation prior to use. All solvents were purchased from Sigma-Aldrich or Fisher Scientific.

Unless stated otherwise, all NMR spectroscopic analyses were recorded at 298 K using a Bruker Avance III 500.12 MHz spectrometer with 1H NMR spectra run at 500.12 MHz, and 29Si NMR spectra at 99.37 MHz. The 1H NMR spectra were referenced internally using residual solvent signals and are reported relative to external tetramethylsilane. Chemical shifts are quoted in ppm and coupling constants in Hz.

Experimental details

Potassium and sodium metal, 2-tert-butyl-4-methylphenol, 2,4-bis(dimethylbenzyl)phenol, terephthalaldehyde and isophthaldehyde were purchased from Sigma-Aldrich and used as received. KN(SiMe3)2,30 [Ca(N(SiMe3)2]2,31 UI4(dioxane)2 and (((Me3Si)2)N)2U[κ-(N=S)SiMe3]2,32 KN(SiMe3)2,30 [Ca(N(SiMe3)2]2,31 UI4·(dioxane)2,32 and KCa were synthesised as previously described in literature procedures.34

H4Lp*

Analogous procedure to that used to synthesise H4Lp. 82% yield.

1H NMR (C6D6, 600 MHz) δ 7.34–7.28 (Aromatic H, 12H), 7.20 (Aromatic H, 8H), 7.12 (Aromatic H, 8H), 7.07 (Aromatic H, 4H), 7.03 (Aromatic H, 4H), 6.99 (Aromatic H, 8H), 6.96–6.90 (Aromatic H, 4H), 6.89 (Aromatic H, 4H), 5.98 (Ar=CH2, 2H), 4.48 (ArOH, 4H), 1.65 (CH3, 24H), 1.47 (CH3, 24H).

Elemental analysis: C 82.71%, H 8.81% calculated. C 82.83%, 8.92% found.

Mass Spectrometry: (ESI) m/z 777.4850 [Lp⁺ + Na⁺].

1P (U2Lp)∗

Two 100 mL Schlenk flasks were charged respectively with [CaN(SiMe3)2]2 (0.978 g, 1.36 mmol) and H4Lp (1.024 g, 1.36 mmol) and equipped with stirrer bars. 30 mL of 1,4-dioxane was added to both solids to provide off-white solutions. The solutions were combined into one Schlenk with vigorous stirring and stirred for an hour at room temperature, to provide an off-white suspension. To a 250 mL Schlenk flask containing U4I12(OCH2H4O)2 and a stirrer bar, 100 mL of 1,4-dioxane was added to yield a slightly turbid red solution. The Ca2Lp suspension generated in situ was added to the U4I12(OCH2H4O)2 solution with vigorous stirring. Once the addition was complete, the reaction was left to stir for 48 hours to yield a light green suspension. The green brown solution was filtered and isolated by filtration and washed with MeCN. The resulting colourless solid was dried under vacuum at 65 °C overnight and stored in a glove box. Yield 22.9 g (82%).

1H NMR (C6D6, 500 MHz) δ 7.09 (Aryl oxide H, s, 4H), 7.06–6.96 (Aromatic H, m, 4H), 6.68 (Aromatic H, s, 4H), 5.47 (Ar=CH2, s, 2H), 4.90 (ArOH, s, 4H), 2.05 (CH2, s, 12H), 1.43 (Bu H, s, 36H).

13C NMR (C6D6, 126 MHz) δ 151.1, 141.8, 137.6, 130.7, 129.6, 128.4, 128.0 (Aromatic C), 47.6 (Ar=CH2), 34.5 (C(CH3)3), 29.61 (C(CH3)3), 20.73 (CH3).

Elemental analysis: C 82.71%, H 8.81% calculated. C 82.83%, 8.92% found.

Mass Spectrometry: (ESI) m/z 1441.7983 [Lp⁺ + Na⁺].
1\(^{1s}\) (U\(_2\)I\(_4\)LP\(^{2+}\))

Made analogously to U\(_2\)I\(_4\)LP. Green plates suitable for X-ray diffraction were grown from slow evaporation of a THF solution.

\(^{1}\)H NMR (\(d_8\)-THF, 329 K, 500 MHz) \(\delta\) 7.13 (Aryl H, 2H), 7.08 (Aryl H, 8H), 6.98 (Aryl H, 4H), 6.94 (Aryl H, 4H), 6.84 (Aryl H, 2H), 6.69 (Aryl H, 2H), 6.57 (Aryl H, 8H), 6.47 (Aryl H, 4H), 6.42 (Aryl H, 2H), 6.56 (Aryl H, 4H), 2.31 (Ar,CH\(_3\), 1H), 2.21 (Ar,CH\(_3\), 1H), 1.45 (CH\(_3\), 12H), 1.31 (Aryl H, 4H), 1.18 (CH\(_3\), 12H), 1.02 (Aryl H, 4H), 0.78 (Aryl H, 4H).

Elemental analysis: C 53.62%, H 5.02% found.

A Schlenk flask was charged with H\(_4\)LP (100 mg, 0.133 mmol) and KN\(^+\) (1.06 g, 5.30 mmol) and equipped with a stirrer bar. THF was added and the yellow solution was stirred for 1 hour at room temperature. To this solution, U\(_2\)(OC\(_6\)H\(_4\)O\(_2\))\(_2\) \((2.44 \text{ g, } 2.65 \text{ mmol})\) in THF was added by cannula transfer from a separate Schlenk flask. The resulting dark green solution was stirred at room temperature for 48 hours, yielding a pale green suspension. The colourless precipitate was removed by filtration and the solvent was removed under reduced pressure giving a light green solid. \(\text{(1.87 g, 82%). Green plate crystals suitable for single crystal X-ray analysis were grown from slow evaporation of a THF solution.}

\(^{1}\)H NMR (\(d_8\)-THF, 329 K, 500 MHz) \(\delta\) 7.13 (Aryl H, 2H), 7.08 (Aryl H, 8H), 6.98 (Aryl H, 4H), 6.94 (Aryl H, 4H), 6.84 (Aryl H, 2H), 6.69 (Aryl H, 2H), 6.57 (Aryl H, 8H), 6.47 (Aryl H, 4H), 6.42 (Aryl H, 2H), 6.56 (Aryl H, 4H), 2.31 (Ar,CH\(_3\), 1H), 2.21 (Ar,CH\(_3\), 1H), 1.45 (CH\(_3\), 12H), 1.31 (Aryl H, 4H), 1.18 (CH\(_3\), 12H), 1.02 (Aryl H, 4H), 0.78 (Aryl H, 4H).

Elemental analysis: C 53.62%, H 5.02% calculated.

A Schlenk flask was charged with H\(_4\)LM \((1.00 \text{ g, } 1.32 \text{ mmol})\) and KN\(^+\) \((1.06 \text{ g, } 5.30 \text{ mmol})\) and equipped with a stirrer bar. THF was added and the yellow solution was stirred for 1 hour at room temperature. To this solution, U\(_2\)(OC\(_6\)H\(_4\)O\(_2\))\(_2\) \((2.44 \text{ g, } 2.65 \text{ mmol})\) in THF was added by cannula transfer from a separate Schlenk flask. The resulting dark green solution was stirred at room temperature for 48 hours, yielding a pale green suspension. The colourless precipitate was removed by filtration and the solvent was removed under reduced pressure giving a light green solid. \(\text{(1.87 g, 82%). Green plate crystals suitable for X-ray diffraction were grown from slow evaporation of concentrated benzene or THF solutions at room temperature.}

\(^{1}\)H NMR (\(d_8\)-THF, 329 K, 500 MHz) \(\delta\) 7.13 (Aryl H, 2H), 7.08 (Aryl H, 8H), 6.98 (Aryl H, 4H), 6.94 (Aryl H, 4H), 6.84 (Aryl H, 2H), 6.69 (Aryl H, 2H), 6.57 (Aryl H, 8H), 6.47 (Aryl H, 4H), 6.42 (Aryl H, 2H), 6.56 (Aryl H, 4H), 2.31 (Ar,CH\(_3\), 1H), 2.21 (Ar,CH\(_3\), 1H), 1.45 (CH\(_3\), 12H), 1.31 (Aryl H, 4H), 1.18 (CH\(_3\), 12H), 1.02 (Aryl H, 4H), 0.78 (Aryl H, 4H).

Elemental analysis: C 53.62%, H 5.02% calculated.

2\(^{2+}\) (U\(_2\)N\(_4\)LP\(^{2+}\))

Made by an analogous procedure. Green plates isolated in 65% yield. Single crystals suitable for X-ray diffraction were grown by diffusion of hexanes into a THF solution of \(2^+\).

\(^{1}\)H NMR (\(C_6D_6\), 500 MHz) \(\delta\) 41.19 (Aromatic H, 2H), 31.62 (Aromatic H, 1H), 27.73 (Aromatic H, 1H), 16.71 (Aroyl oxide H, 4H), 3.90 (Aroyl oxide H, 4H), 1.50 (CH\(_3\), 12H), −3.03 (Ar,CH\(_2\), 2H), −9.76 (t-BuH, 36H), −18.51 (SiCH\(_3\), 72H) ppm; \(^{29}\)Si NMR (\(C_6D_6\), 99.4 MHz) \(\delta\) −230.8 (Me, Si).

Elemental analysis: C 48.65%, H 7.23%, N 3.00% calculated. C 48.66%, H 6.91%, N 2.78% found.

3\(^{3+}\) (U\(_2\)N\(_4\)LM\(^{3+}\))

A Schlenk flask was charged with U\(_2\)N\(_4\)LM \((274 \text{ mg, } 0.223 \text{ mmol})\) and K\(_2\) \((64 \text{ mg, } 0.447 \text{ mmol})\) and equipped with a stirrer bar. Toluene was added and the resulting dark green solution was stirred for 16 hours at room temperature, turning dark purple. The toluene was removed under reduced pressure and the product was extracted into heptane. The dark purple product was obtained as a powder following removal of volatiles under reduced pressure, \((0.31 \text{ g, } 74%)\).

\(^{1}\)H NMR (\(C_6D_6\), 500 MHz) \(\delta\) 21.32 (Aromatic H, 8H), 19.97 (Aroyl oxide H, 4H), 14.62 (Aromatic H, 1H), 11.85 (Aroyl oxide H, 4H), 7.50 (Aroyl oxide H, 4H), 2.11 (CH\(_3\), 12H), 0.89 (Ar,CH\(_2\), 2H), −7.94 (s, 36H, t-BuH), −13.33 (s, 36H, SiCH\(_3\)) ppm; \(^{29}\)Si NMR (\(C_6D_6\), 99.4 MHz) \(\delta\) −99.93 (Me, Si).

Elemental analysis: C 49.66%, H 6.38%, N 1.81% calculated. C 49.66%, H 6.38%, N 1.81% found.

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Notes and references
