

Supporting Information for

Understanding Geometric Preferences in Uranium(VI) Mixed Tris(imido) Systems

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General Considerations.

All air- and moisture-sensitive manipulations were performed using standard Schlenk techniques or in an MBraun inert atmosphere drybox with an atmosphere of purified nitrogen. The MBraun drybox was equipped with a cold well designed for freezing samples in liquid nitrogen as well as two -35 °C freezers for cooling samples and crystallizations. Solvents for sensitive manipulations were dried and deoxygenated using literature procedures with a Seca solvent purification system.[1] Benzene-d₆ was purchased from Cambridge Isotope Laboratories. Deuterated solvents were degassed by three freeze-pump-thaw cycles and stored over molecular sieves and sodium. ^{Mes}PDI^{Me},[2] [(^{Mes}PDI^{Me})U(THF)]₂,[3] and aryl azides[4] were prepared according to literature procedures.

¹H NMR spectra were recorded on a Varian Inova 300 spectrometer at 299.992 MHz. All chemical shifts are reported relative to the peak for SiMe₄, using ¹H (residual) chemical shifts of the solvent as a secondary standard. The spectra for paramagnetic molecules were obtained by using an acquisition time of 0.5 s; thus, the peak widths reported have an error of ±2 Hz. For paramagnetic molecules, the ¹H NMR data are reported with the chemical shift, followed by the peak width at half height in Hertz, the integration value, and, where possible, the peak assignment. Elemental analyses were performed by Midwest Microlab (Indianapolis, IN).

Preparation of (^{Mes}PDI^{Me})U(NDipp)₂(NMe_s) (2-(Dipp)₂(Me_s)). A 20 mL scintillation vial was charged with **1-THF** (0.250 g, 0.177 mmol) and dissolved in THF (10 mL). To this stirring dark brown solution was slowly added a THF solution containing two equivalents of 2,6-diisopropylphenylazide (0.144 g, 0.708 mmol) and one equivalent of 2,4,6-trimethylphenylazide (0.057 g, 0.354 mmol) and effervescence of N_{2(g)} was immediately observed. Following addition, the volatiles were removed and the brown solid identified as (^{Mes}PDI^{Me})U(NDippP)₂(NMe_s) (**2-(Dipp)₂(Me_s)**) was collected. (Yield; 0.350 g, 0.301 mmol, 85%) Further purification was accomplished by stirring in cold pentane followed by immediate filtration and collection of solid. X-ray quality single crystals were grown by layering a THF solution with pentane at -35° C.

2-(Dipp)₂(Me_s): ¹H NMR (300 MHz, C₆D₆) δ = 0.71 (d, *J* = 6.0 Hz, 12H, *i*PrCH₃), 1.53 (d, *J* = 6 Hz, 12H, *i*PrCH₃), 1.71 (s, 6H, PDI-*p*-ArCH₃), 2.02 (s, 12H, PDI-*o*-ArCH₃), 2.12 (s, 6H, PDI-CH₃), 3.69 (sept, *J* = 6 Hz, 2H, *i*PrCH), 4.41 (s, 6H, *o*-ArCH₃), 5.86 (s, 3H, *p*-ArCH₃), 5.93 (t, 2H, Ar-CH), 6.72 (s, 4H, PDI-ArH), 7.24 (t, *J* = 6 Hz, 1H, *p*-PyrH), 7.29 (d, *J* = 6 Hz, 2H, *m*-PyrH),

7.79 (d, $J = 6$ Hz, 2H, *m*-ArH), 8.15 (s, 2H, Ar-CH), 8.44 (sept, $J = 6$ Hz, 2H, *i*PrCH). ^{13}C NMR (400 MHz, C_6D_6) $\delta = 12.36, 17.94, 18.69, 18.85, 18.93, 20.66, 25.51, 25.68, 26.58, 109.07, 110.48, 117.86, 121.35, 123.18, 126.48, 129.62, 129.98, 136.80, 138.79, 141.67, 152.69, 156.69, 156.89, 158.61, 163.54, 170.76, 174.67, 177.54$. Elemental Analysis for $\text{C}_{60}\text{H}_{76}\text{N}_6\text{U}$: calc. C: 64.38; H: 6.84; N: 7.51, found: C: 61.25; H: 7.04; N: 7.04. Complete combustion was not attainable. Therefore, the found carbon percentage is low.

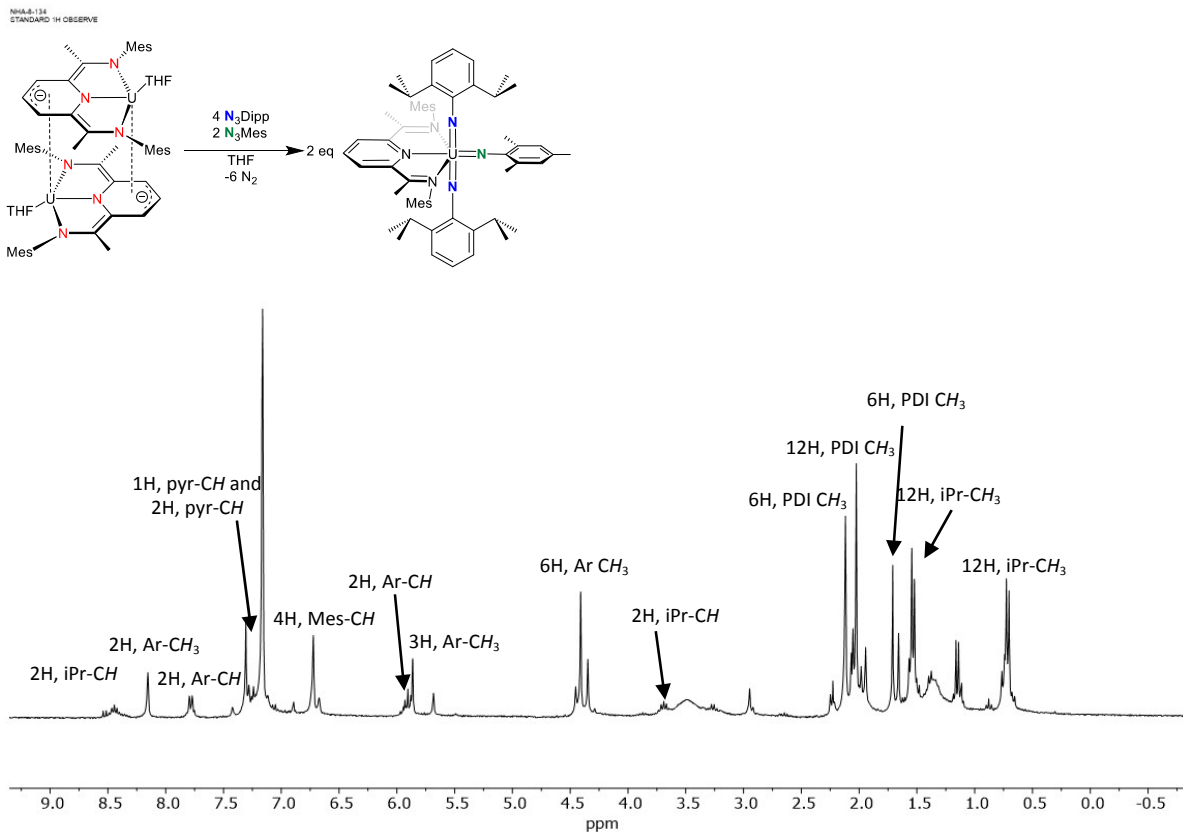


Figure S1. ^1H NMR spectrum (ambient temperature, C_6D_6) of $\text{MesPDI}^{\text{Me}}\text{U}(\text{NDipp})_2(\text{NMes})$ ($2\text{-(Dipp)}_2(\text{Mes})$).

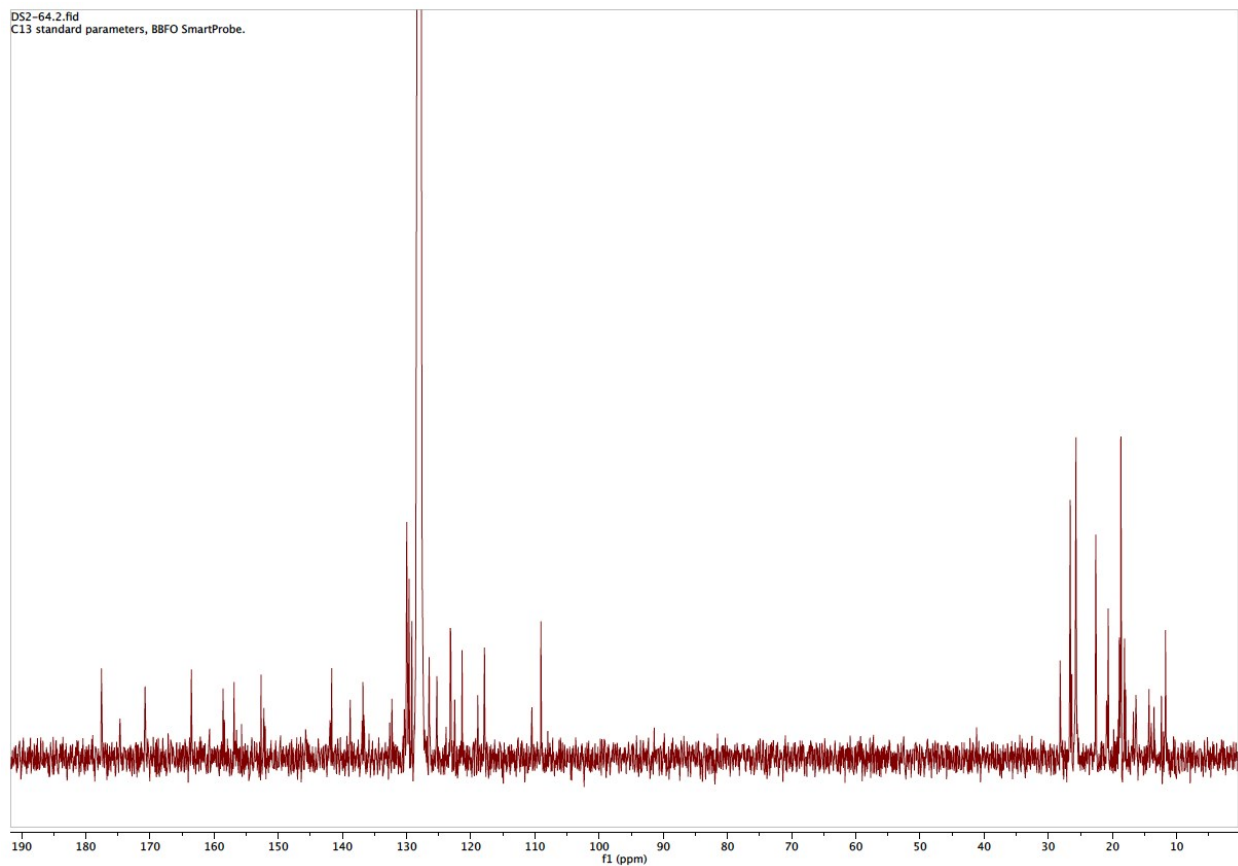


Figure S2. ^{13}C NMR spectrum (ambient temperature, C_6D_6) of $^{\text{Mes}}\text{PDI}^{\text{Me}}\text{U}(\text{NDipp})_2(\text{NMes})(2\text{-(Dipp)}_2(\text{Mes}))$.

Preparation of $(^{\text{Mes}}\text{PDI}^{\text{Me}})\text{U}(\text{NDipp})_2(\text{NDetp})$ (2-(Dipp)₂(Detp)**).** A 20 mL scintillation vial was charged with **1-THF** (0.250 g, 0.177 mmol) and dissolved in THF (10 mL). To this stirring dark brown solution was slowly added a THF solution containing two equivalents of 2,6-diisopropylphenylazide (0.144 g, 0.708 mmol) and one equivalent of 2,6-diethylphenylazide (0.062 g, 0.354 mmol) and effervescence of $\text{N}_{2(\text{g})}$ was immediately observed. Following addition, the volatiles were removed and the brown solid identified as $(^{\text{Mes}}\text{PDI}^{\text{Me}})\text{U}(\text{NDipp})_2(\text{NDetp})$ (**2-(Dipp)₂(Detp)**) was collected. (Yield; 0.361 g, 0.319 mmol, 90%) Further purification was accomplished by stirring in cold pentane followed by immediate filtration and collection of solid. X-ray quality single crystals were grown by layering a THF solution with pentane at -35°C .

2-(Dipp)₂(Detp): ^1H NMR (300 MHz, C_6D_6) δ = 0.75 (d, 12H, *iPrCH*₃, J = 7.0 Hz), 1.19 (t, 6H, $-\text{CH}_2\text{CH}_3$, J = 7.3 Hz), 1.51 (d, 12H, *iPrCH*₃, J = 6.7 Hz), 1.71 (s, 6H, *Mes-CH*₃), 2.05 (s, 12H, *Mes-CH*₃), 2.18 (s, 6H, *PDI-CH*₃), 3.71 (m, 2H, *iPr-CH*), 5.50 (q, 4H, $-\text{CH}_2\text{CH}_3$, J = 7.3 Hz), 5.87 (t, 2H, *Ar-CH*, J = 7.5 Hz), 6.74 (s, 4H, *Ar-CH*), 7.26 (s, 1H, *Ar-CH*), 7.30 (m, 5H, *ArCH* + *Ar-CH*), 7.74 (d, 2H, *Ar-CH*, J = 7.2 Hz), 8.34 (sept, 2H, *iPr-CH*, J = 6.7 Hz), 8.47 (d, 2H, *pyr-CH*, J = 7.41 Hz). ^{13}C NMR (400 MHz, C_6D_6) δ = 14.94, 16.57, 17.53, 18.72, 18.89, 20.85, 22.60, 25.70, 26.65, 106.43, 107.09, 117.73, 121.22, 123.46, 126.56, 126.67, 129.50, 130.17, 136.55, 138.72, 152.72, 156.82, 163.16, 171.39, 180.23. Elemental Analysis for $\text{C}_{61}\text{H}_{78}\text{N}_6\text{U}$: calc. C: 64.65; H: 6.94; N: 7.42, found: C: 60.59; H: 6.82; N: 7.24. Complete combustion was not attainable. Therefore, the found carbon percentage is low.

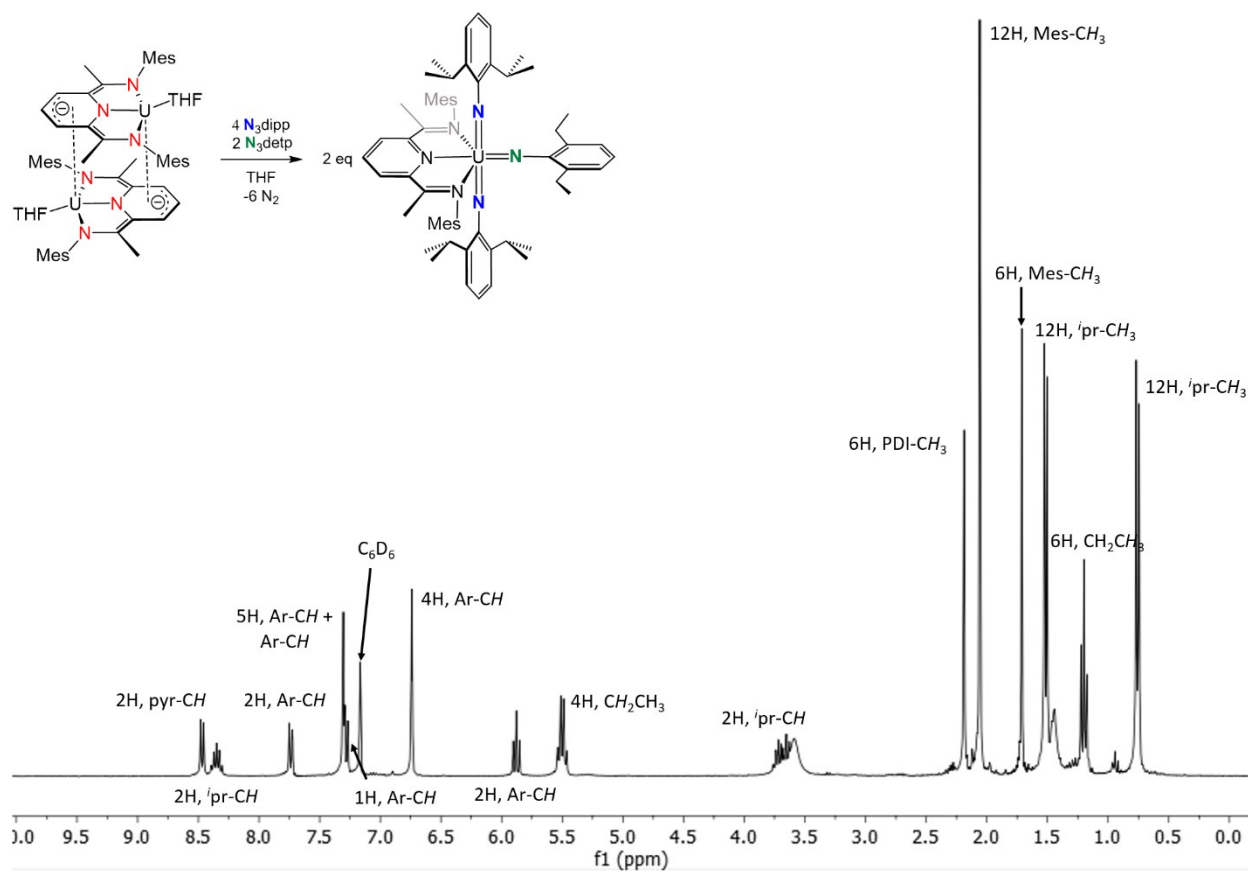


Figure S3. ¹H NMR spectrum (C₆D₆, ambient temperature) of 2-(Dipp)₂(Detp).

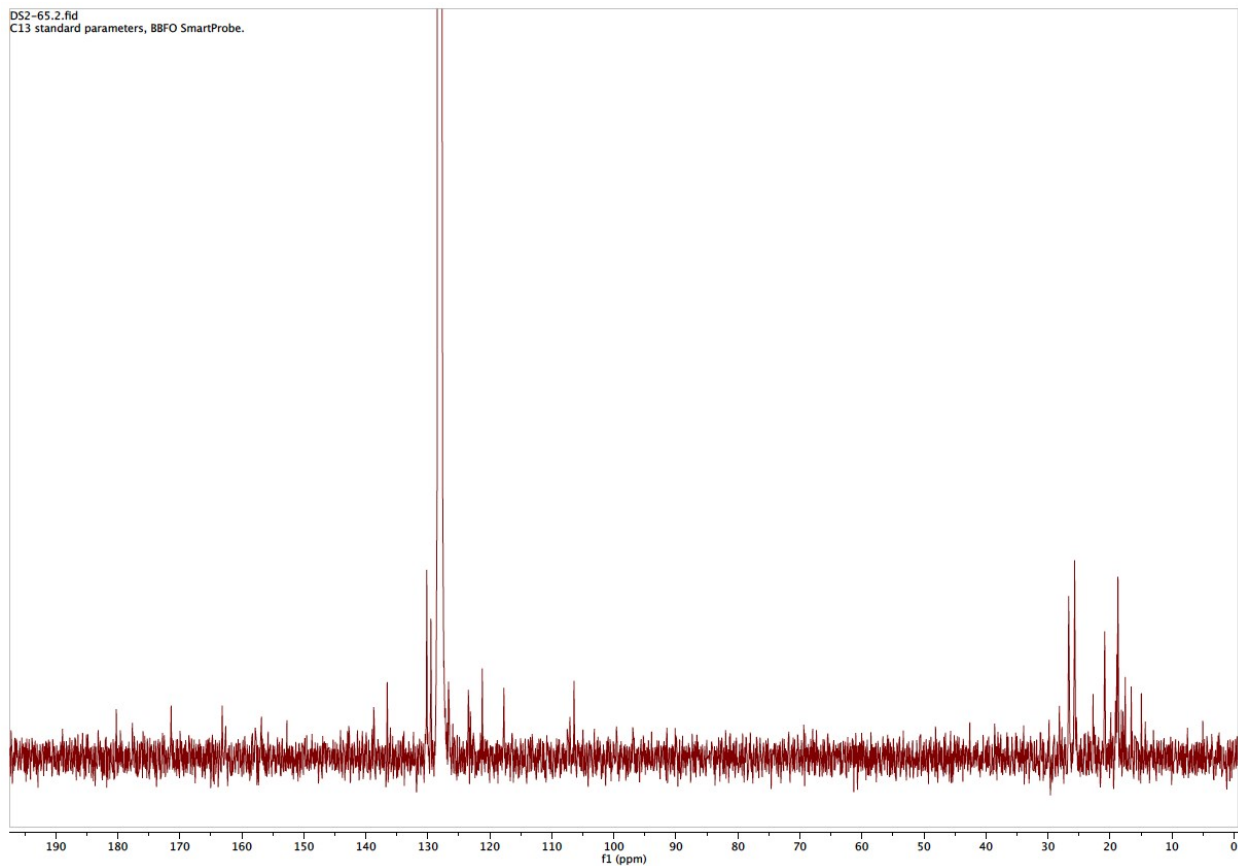


Figure S4. ^{13}C NMR spectrum (C_6D_6 , ambient temperature) of **2-(Dipp) $_2$ (Detp)**.

Preparation of ^{Mes}PDI^{Me}U(N-Detp)₃ (2-Detp).

Following a previously published method,^[3] a 20-mL scintillation vial was charged with **1-THF** (0.250 g, 0.177 mmol), THF (8 mL), and a stir bar. While stirring, this dark brown solution was treated with six equivalents of 2,6-diethylphenylazide (0.184 g, 1.06 mmol). Upon azide addition, effervescence of N_{2(g)} was immediately observed. After stirring, the volatiles were removed *in vacuo*. Further purification was accomplished by stirring the brown residue in cold (-35 °C) *n*-pentane followed by immediate filtration, leaving a brown solid identified as (^{Mes}PDI^{Me})U(NDetp)₃ (Yield: 0.220 g, 0.280 mmol, 58%). X-ray quality single crystals were grown by layering a THF solution with pentane at -35° C.

2-Detp: ¹H NMR (300 MHz, C₆D₆) δ = 0.65 (t, 6H, -CH₂CH₃, *J* = 7.3 Hz), 1.29 (t, 6H, -CH₂CH₃, *J* = 7.4 Hz), 1.46 (t, 6H, -CH₂CH₃, *J* = 7.1 Hz), (1.66 (s, 6H, Mes-CH₃), 2.01 (s, 12H, Mes-CH₃), 2.17 (s, 6H, PDI-CH₃), 2.76 (q, 4H, -CH₂H₃, *J* = 7.4 Hz), 3.63 (t, 1H, Ar-CH, *J* = 7.2 Hz), 5.01 (q, 4H, -CH₂CH₃, *J* = 7.3 Hz), 5.51 (q, 4H, -CH₂CH₃, *J* = 7.3 Hz), 5.86 (t, 2H, Ar-CH, *J* = 7.3 Hz), 6.72 (s, 4H, Mes-CH), 7.25 (t, 1H, pyr-CH, *J* = 7.3 Hz), 7.69 (d, 2H, Ar-CH, *J* = 7.2 Hz), 8.44 (d, 2H, pyr-CH, *J* = 7.3 Hz). ¹³C NMR (400 MHz, C₆D₆) δ = 16.39, 16.76, 17.81, 18.24, 18.29, 18.83, 20.78, 22.94, 24.07, 107.86, 120.66, 123.22, 126.62, 129.55, 130.12, 136.49, 139.06, 142.57, 147.97, 156.47, 157.82, 159.63, 171.46, 178.53, 188.60. Elemental Analysis for C₅₇H₇₀N₆U: calc. C: 63.55; H: 6.55; N: 7.80, found: C: 55.63; H: 5.79; N: 7.02. Complete combustion was not attainable. Therefore, the found percentages are low.

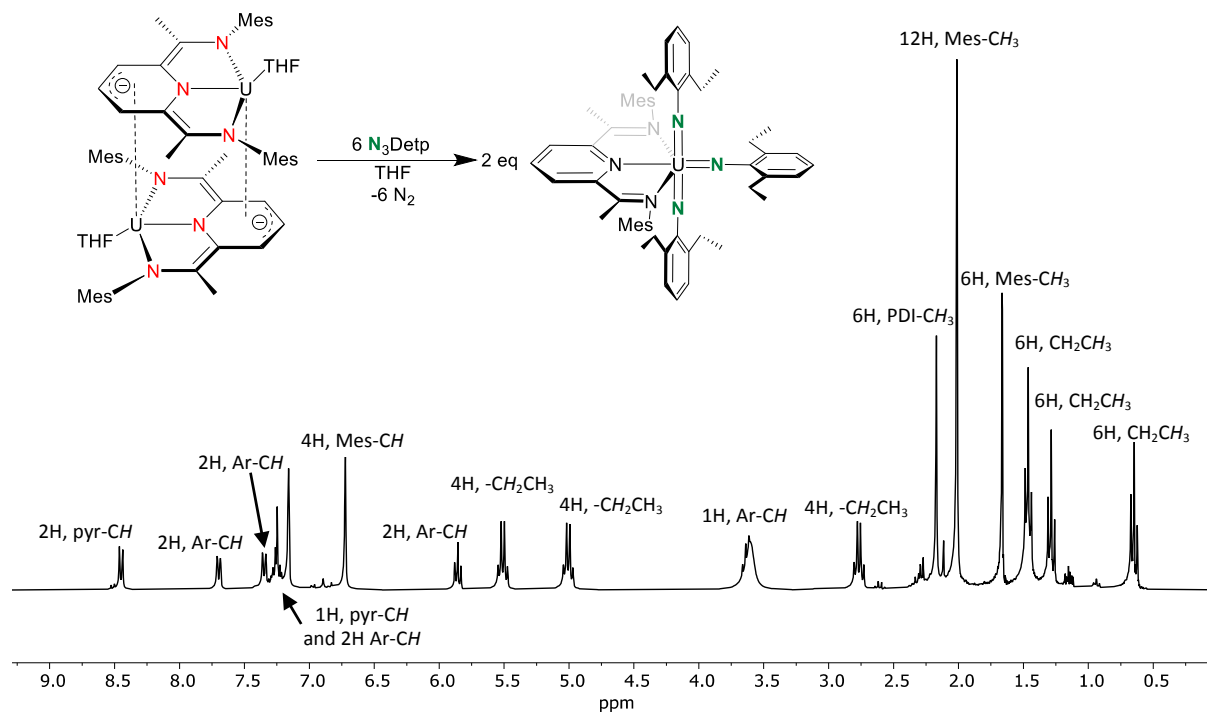


Figure S5. 1H NMR spectrum (ambient temperature, C_6D_6) of **2-Detp**.

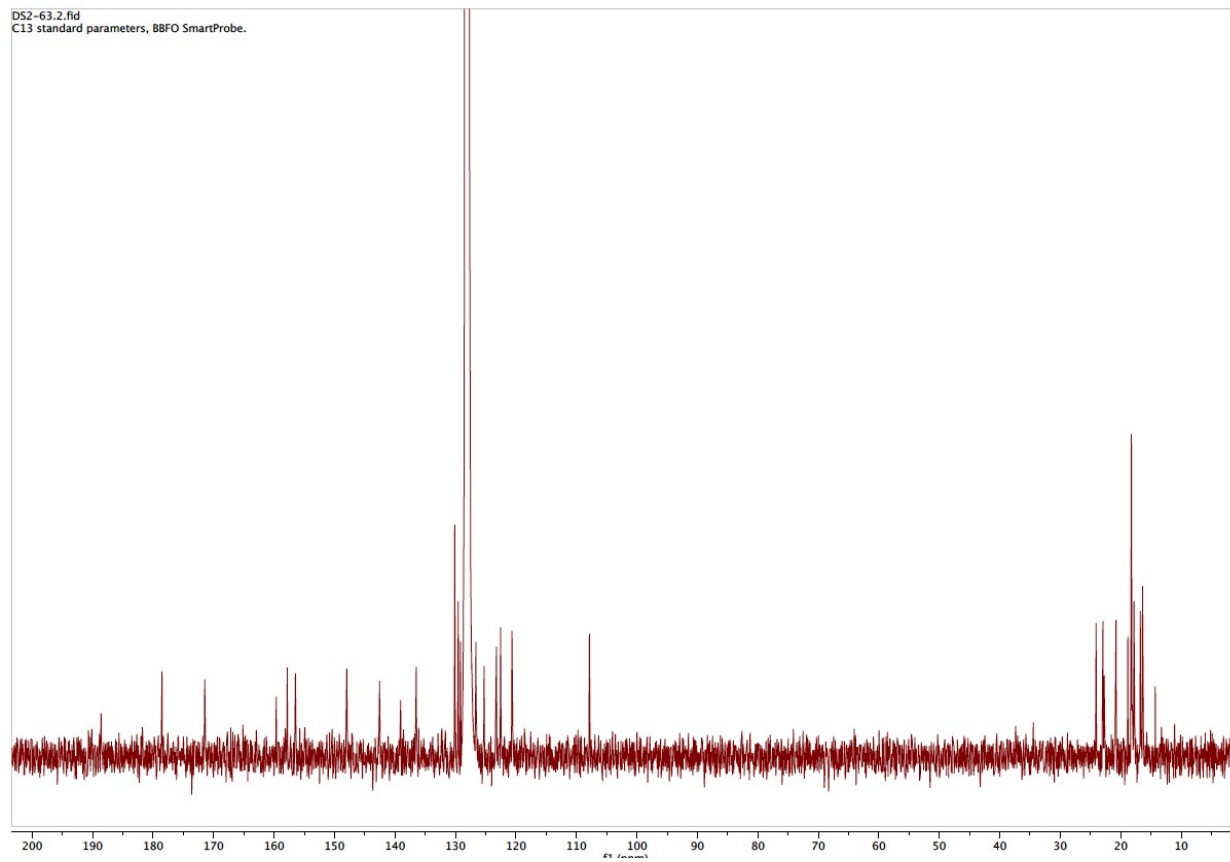


Figure S6. ^{13}C NMR spectrum (ambient temperature, C_6D_6) of **2-Dehp**.

Reaction between $[(\text{PDI})\text{U}(\text{THF})]_2$, 4 eq N_3Detp and 2 eq N_3Dipp :

AR-1-194_2_washed
AR-1-194_2 filtrate

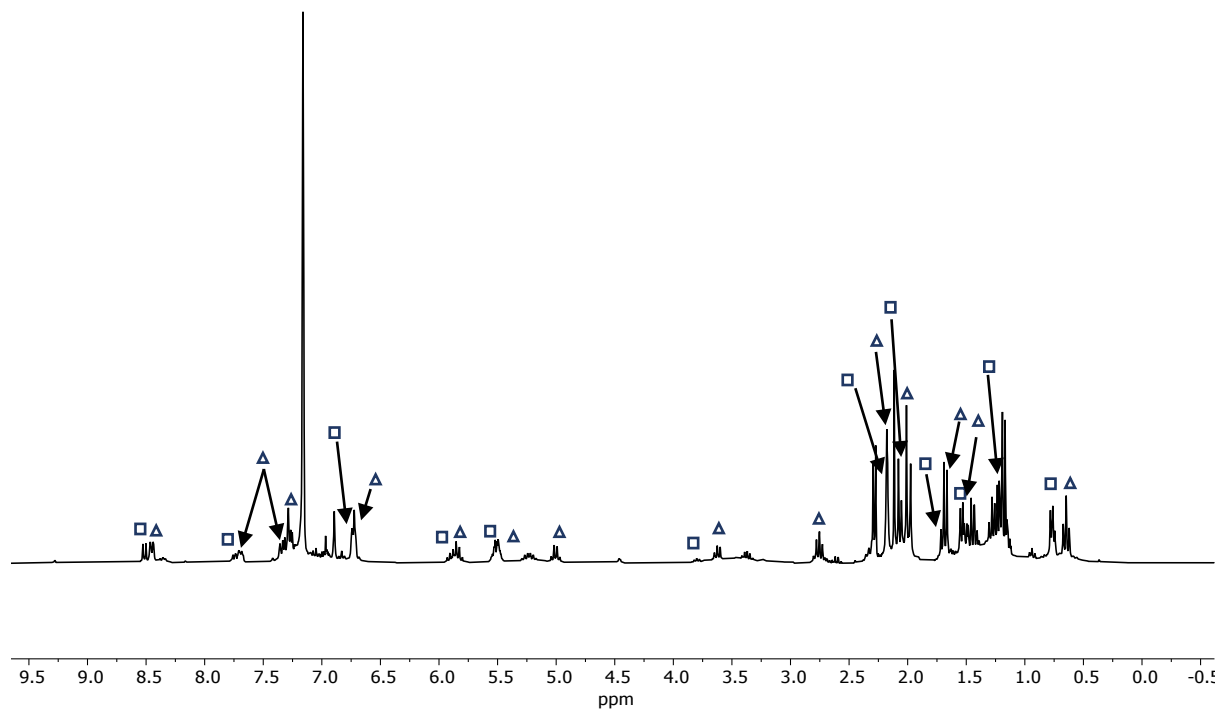


Figure S7. ^1H NMR spectrum (ambient temperature, C_6D_6) of the reaction between $[(\text{PDI})\text{U}(\text{THF})]_2$, 4 eq N_3Detp and 2 eq N_3Dipp . Peaks corresponding to **2-Detp** are indicated by triangles and those corresponding to **2-(Dipp)₂(Detp)** by squares.

Table S1. Selected Bond Metrics (in Å or °)

	2-Detp	2-(Dipp)₂(Mes)	2-(Dipp)₂(Detp)
U1-N1	2.599(7)	2.585(8)	2.564(3)
U1-N2pyr	2.587(7)	2.569(9)	2.589(3)
U1-N3	2.548(8)	2.522(7)	2.558(3)
U1-N4	1.989(7)	1.967(7)	1.987(3)
U1-N5	1.970(7)	1.996(6)	1.988(3)
U1-N6	2.030(7)	2.028(8)	2.033(3)
C1-C2	1.515(12)	1.495(14)	1.502(5)
C2-N1	1.288(11)	1.274(12)	1.283(5)
C2-C3	1.494(12)	1.491(12)	1.486(5)
C3-N2	1.358(10)	1.348(12)	1.350(4)
C3-C4	1.402(12)	1.396(12)	1.398(5)
C7-N2	1.329(11)	1.340(11)	1.343(5)
C7-C8	1.501(11)	1.480(13)	1.488(5)
C7-C6	1.380(11)	1.408(13)	1.389(5)
C8-N3	11.285(11)	1.298(12)	1.284(5)
C8-C9	1.507(11)	1.490(13)	1.493(5)
C4-C5	1.389(13)	1.392(14)	1.383(5)
C5-C6	1.402(12)	1.356(14)	1.387(5)
N4-U1-N5	168.4(3)	167.9(3)	167.13(12)
N4-U1-N6	96.6(3)	95.9(3)	96.75(12)
N5-U1-N6	95.0(3)	96.2(3)	96.11(12)

Single Crystal Determinations

Single crystals of **2-(Detp)** suitable for X-ray diffraction were coated with poly(isobutylene) oil in a glovebox and quickly transferred to the goniometer head of the specified instrument Rigaku R-axis curved image plate diffractometer. Crystals were transferred to the goniometer head of a Rigaku R-axis curved image plate diffractometer equipped with MicroMax002+ high-intensity copper X-ray source with confocal optics and examined with Cu K α radiation ($\lambda = 1.54184 \text{ \AA}$). In a similar manner, a single crystal of **2-(Dipp)₂(Mes)** was transferred to the goniometer head of a Nonius Kappa CCD diffractometer equipped with a graphite crystal and incident beam monochromator and examined with Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$). Single crystal of **2-(Dipp)₂(Detp)** were transferred to the goniometer head of a Bruker Quest diffractometer equipped with a single crystal curved graphite incident beam monochromator and a Photon100 CMOS area detector. Examination and data collection were performed with Cu K α radiation ($\lambda = 1.54184 \text{ \AA}$).

Data were collected at low temperature using the Nonius Collect^[5] or dtrek option of CrystalClear software.^[6] Data from the Bruker Quest instrument were collected, reflections were indexed and processed, and the files scaled and corrected for absorption using APEX3.^[7] All data sets were processed using HKL3000^[8] and data were corrected for absorption and scaled using Scalepack.^[8] The space groups were assigned using XPREP from the Shelxtl suite of programs^[9] and the structures were solved by direct methods with SHELXS^[9] and refined by full matrix least squares against F^2 with all reflections using the graphical user interface ShelXle^[10] for the refinement program SHELXL.^[11] H atoms attached to carbon and boron atoms were positioned geometrically and constrained to ride on their parent atoms, with carbon hydrogen bond distances of 0.95 Å for aromatic C-H, 1.00, 0.99 and 0.98 Å for aliphatic C-H, CH₂ and CH₃ moieties, respectively. Methyl H atoms were allowed to rotate but not to tip to best fit the experimental electron density. $U_{\text{iso}}(\text{H})$ values were set to a multiple of $U_{\text{eq}}(\text{C/B})$ with 1.5 for CH₃, and 1.2 for C-H, CH₂ units, respectively. These data have been deposited in the Cambridge Crystallographic Data Centre (CCDC) [1996212-1996214] and can be accessed free of charge at <https://www.ccdc.cam.ac.uk/structures/>.

Local name: AR-1-186c

CCDC: 1996213

Table S2. Experimental details for (^{Mes}PDI^{Me})U(N-Detp)₃ (**2-Detp**)

Crystal data	
Chemical formula	C ₅₇ H ₇₀ N ₆ U
<i>M</i> _r	1077.22
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	14.6346 (5), 21.6509 (7), 15.5875 (5)
β (°)	90.829 (3)
<i>V</i> (Å ³)	4938.4 (3)
<i>Z</i>	4
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	9.57
Crystal size (mm)	0.23 × 0.20 × 0.05
Data collection	
Diffractometer	Rigaku Rapid II curved image plate diffractometer
Absorption correction	Multi-scan SCALEPACK ^[8]
<i>T</i> _{min} , <i>T</i> _{max}	0.217, 0.646
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	39257, 8382, 7047

R_{int}	0.055
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.067, 0.210, 1.15
No. of reflections	8382
No. of parameters	591
H-atom treatment	H-atom parameters constrained
	$w = 1/[\sigma^2(\text{Fo}^2) + (0.1495\text{P})^2 + 4.6136\text{P}]$ where $\text{P} = (\text{Fo}^2 + 2\text{Fc}^2)/3$
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e \AA^{-3})	3.43, -3.31

Computer programs: *CrystalClear-SM Expert 2.1* b32,^[6] *HKL-3000*,^[8] *SHELXS97*,^[9] *SHELXL2014/7*,^[11] *SHELXLE Rev816*.^[10]

Local name: nha23

CCDC: 1996212

Table S3. Experimental details for (^{Mes}PDI^{Me})U(N-Dipp)₂(N-Mes) (**2-(Dipp)₂(Mes)**)

Crystal data	
Chemical formula	C ₆₀ H ₇₆ N ₆ U·C ₅ H ₁₂
<i>M</i> _r	1191.44
Crystal system, space group	Orthorhombic, <i>Pna</i> 2 ₁
Temperature (K)	150
<i>a</i> , <i>b</i> , <i>c</i> (Å)	23.3078 (12), 14.5446 (4), 17.1713 (5)
<i>V</i> (Å ³)	5821.1 (4)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	2.83
Crystal size (mm)	0.70 × 0.50 × 0.50
Data collection	
Diffractometer	Nonius KappaCCD
Absorption correction	Multi-scan <i>SCALEPACK</i> ^[8]
<i>T</i> _{min} , <i>T</i> _{max}	0.180, 0.243
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	14104, 14104, 8340
<i>R</i> _{int}	0.067
(sin θ/λ) _{max} (Å ⁻¹)	0.675

Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.043, 0.084, 0.92
No. of reflections	14104
No. of parameters	671
No. of restraints	1
H-atom treatment	H-atom parameters constrained
	$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{\max}, \Delta\rho_{\min}$ (e Å ⁻³)	1.57, -1.10
Absolute structure	Refined as an inversion twin.
Absolute structure parameter	0.429 (8)

Computer programs: Nonius Collect,^[5] *HKL-2000*,^[8] *SIR2004* (Burla, *et al.*, 2005), *SHELXL2018/3*,^[11] *SHELXLE Rev946*.^[10]

Local name: CTAR2

CCDC: 1996214

Table S4. Experimental details for $(^{\text{Mes}}\text{PDI}^{\text{Me}})\text{U}(\text{NDipp})_2(\text{NDetp})$ (**2-(Dipp)₂(Detp)**)

Crystal data	
Chemical formula	$\text{C}_{65}\text{H}_{86}\text{N}_6\text{OU}$
M_r	1205.42
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	11.3843 (4), 23.5278 (9), 22.0095 (8)
β (°)	100.3112 (12)
V (Å ³)	5800.0 (4)
Z	4
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	8.22
Crystal size (mm)	0.28 × 0.19 × 0.09
Data collection	
Diffractometer	Bruker AXS D8 Quest CMOS diffractometer
Absorption correction	Multi-scan Apex3 v2016.9-0 ^[7]
$T_{\text{min}}, T_{\text{max}}$	0.492, 0.754
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	523286, 12482, 12224

R_{int}	0.046
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.638
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.030, 0.070, 1.13
No. of reflections	12482
No. of parameters	677
H-atom treatment	H-atom parameters constrained
	$w = 1/[\sigma^2(F_o^2) + (0.017P)^2 + 28.462P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e \AA^{-3})	1.67, -2.05

Computer programs: Apex3 v2016.9-0,^[7] SAINT V8.37A,^[12] SHELXS97,^[9] SHELXL2014/7,^[11] SHELXLE Rev714.^[10]

References

- [1] A. B. Pangborn, M. A. Giardello, R. H. Grubbs, R. K. Rosen, F. J. Timmers, *Organometallics* **1996**, *15*, 1518-1520.
- [2] G. J. P. Britovsek, M. Bruce, V. C. Gibson, B. S. Kimberley, P. J. Maddox, S. Mastroianni, S. J. McTavish, C. Redshaw, G. A. Solan, S. Strömberg, A. J. P. White, D. J. Williams, *Journal of the American Chemical Society* **1999**, *121*, 8728-8740.
- [3] N. H. Anderson, S. O. Odoh, Y. Yao, U. J. Williams, B. A. Schaefer, J. J. Kiernicki, A. J. Lewis, M. D. Goshert, P. E. Fanwick, E. J. Schelter, J. R. Walensky, L. Gagliardi, S. C. Bart, *Nat Chem* **2014**, *6*, 919-926.
- [4] K. Barral, A. D. Moorhouse, J. E. Moses, *Org Lett* **2007**, *9*, 1809-1811.
- [5] Nonius, Delft, The Netherlands, **1998**.
- [6] Rigau Corporation, The Woodlands, TX, **1998**.
- [7] Bruker AXS Inc, Madison, WI, **2016**.
- [8] Z. Otwinowski, W. Minor, *Methods in Enzymology* **1997**, *276*, 307-326.
- [9] G. M. Sheldrick, *Acta Crystallogr A* **2008**, *64*, 112-122.
- [10] C. B. Hübschle, G. M. Sheldrick, B. Dittrich, *J Appl Crystallogr* **2011**, *44*, 1281-1284.
- [11] G. M. Sheldrick, *Acta Crystallogr C Struct Chem* **2015**, *71*, 3-8.
- [12] V8.37A ed., Bruker AXS Inc., Madison, WI, **2015**.