Supporting Information

Controllable Construction of Half-sandwich Octanuclear Complexes Based on Pyridyl-substituted Ligands with Conjugated Centers

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Figure S1. $^1$H NMR (400 MHz, CD$_3$OD- $d_4$) of 4a.
**Figure S2.** $^1$H-$^1$H COSY NMR (400 MHz, CD$_3$OD- $d_4$) of 4a.

**Figure S3.** $^1$H DOSY NMR (400 MHz, CD$_3$OD- $d_4$) of 4a.
Figure S4. $^1$H NMR (400 MHz, CD$_3$OD- $d_4$) of 4b.

Figure S5. $^1$H-$^1$H COSY NMR (400 MHz, CD$_3$OD- $d_4$) of 4b.

Figure S6. $^1$H DOSY NMR (400 MHz, CD$_3$OD- $d_4$) of 4b.
Figure S7. $^1$H NMR (400 MHz, CD$_3$OD- $d_4$) of 4c.

Figure S8. $^1$H-$^1$H COSY NMR (400 MHz, CD$_3$OD- $d_4$) of 4c.
Figure S9. $^1$H DOSY NMR (400 MHz, CD$_3$OD- $d_4$) of 4c.

Figure S10. $^1$H NMR (400 MHz, CD$_3$OD- $d_4$) of 5a.
Figure S11. $^1$H-$^1$H COSY NMR (400 MHz, CD$_3$OD- $d_4$) of 5a.

Figure S12. $^1$H DOSY NMR (400 MHz, CD$_3$OD- $d_4$) of 5a.
Figure S13. $^1$H NMR (400 MHz, CD$_3$OD- $d_4$) of 5b.

Figure S14. $^1$H-$^1$H COSY NMR (400 MHz, CD$_3$OD- $d_4$) of 5b.
**Figure S15.** $^1$H DOSY NMR (400 MHz, CD$_3$OD- $d_4$) of 5b.

**Figure S16.** $^1$H NMR (400 MHz, CD$_3$OD- $d_4$) of 5c.
Figure S17. $^1$H-$^1$H COSY NMR (400 MHz, CD$_3$OD-$d_4$) of 5c.

Figure S18. $^1$H DOSY NMR (400 MHz, CD$_3$OD-$d_4$) of 5c.
Figure S19. $^1$H NMR (400 MHz, CD$_3$OD- $d_4$) of 6.

Figure S20. $^1$H-$^1$H COSY NMR (400 MHz, CD$_3$OD- $d_4$) of 6.
Figure S21. ESI-MS spectra of 4a.
ESI-MS (positive ions) for [M-(H₂O)₂, 5OTf]³⁺: Top (tested) and bottom (Calcd)

Figure S22. ESI-MS spectra of 5a.

ESI-MS (positive ions) for [M-(CH₃OH)₅, 5OTf]³⁺: Top (tested) and bottom (Calcd)
ESI-MS (positive ions) for [M-(CH$_2$OH)$_5$, 6OTf]$^{2+}$: Top (tested) and bottom (Calcd)

**Figure S23.** ESI-MS spectra of 5c.

ESI-MS (positive ions) for [M, 5OTf]$^{3+}$: Top (tested) and bottom (Calcd)
ESI-MS (positive ions) for \([M, 6\text{OTf}]^{2+}\): Top (tested) and bottom (Calcd)

*Figure S24.* ESI-MS spectra of 6.