Supporting Information

Coordination-Driven Self-Assembly of 2D-Metallamacrocycles Using a New Carbazole-Based Dipyridyl Donor: Synthesis, Characterization and C₆₀ Binding Study

Sankarasekaran Shanmugaraju, Vaishali Vajpayee, Sunmi Lee, Ki-Whan Chi, Peter J Stang, and Partha Sarathi Mukherjee

Department of Inorganic and Physical Chemistry, Indian Institute of Science, Bangalore-560 012, India. Fax: 91-80-2360-1552; Tel; 91-80-2293-3352. E-mail: psm@ipc.iisc.ernet.in

Department of Chemistry, University of Ulsan, Ulsan 680-749 Republic of Korea. E-mail: kwchi@ulsan.ac.kr

Department of Chemistry, University of Utah, 315 South 1400 East, Salt Lake City, Utah 84112, United States. E-mail: stang@chem.utah.edu

Figure S1. Infra-red spectrum of bipyridyl donor L and macrocycles 2a–2d.
Figure S2. $^1$H NMR spectrum of dipyridyl donor L recorded in DMSO-$d_6$ with peak assignments.

Figure S3. $^{13}$C NMR spectrum of dipyridyl donor L recorded in DMSO-$d_6$. 
Figure S4. ESI-MS spectrum of dipyridyl donor L recorded in CH$_3$OH.

Figure S5. Solid-state packing view of dipyridyl donor L viewed along crystallographic $b$-axis (color code; C green, H orange, N nitrogen).
**Figure S6.** $^1$H NMR spectra of the macrocycle 2a recorded in CD$_3$NO$_2$ with the peak assignments.

**Figure S7.** $^{31}$P NMR spectra of the macrocycle 2a recorded in CD$_3$NO$_2$ with the peak assignments.
Figure S8. $^1$H NMR spectrum of macrocycle 2b recorded in CD$_3$NO$_2$ with the peak assignments.

Figure S9. $^{31}$P NMR spectrum of macrocycle 2b recorded in CD$_3$NO$_2$.
Figure S10. $^1$H NMR spectrum of macrocycle 2c recorded in DMSO-d$_6$ with the peak assignments.
**Figure S11.** $^1$H NMR spectrum of macrocycle 2d recorded in CDCl$_3$-CD$_3$OD with the peak assignments.

**Figure S12.** $^{31}$P NMR spectrum of macrocycle 2d recorded in CDCl$_3$-CD$_3$OD.
Figure S13. ESI-MS spectrum of macrocycle 2c recorded in CH$_3$CN (inset; experimentally observed isotopic pattern for 2c$^{2+}$ fragment).
Figure S14. ESI-MS spectrum of macrocycle 2d recorded in CH$_3$CN (inset; experimentally observed isotopic pattern for 2d$^{3+}$ fragment).
Figure S15. Solid-state packing diagram of macrocycle 2a viewed along crystallographic $b$-axis (color code; C grey, N blue, O red, H white, Pd magenta, Fe orange, P yellow, F light green).

Table S1. Crystallographic Data and Refinement Parameters of L, 2a.

<table>
<thead>
<tr>
<th></th>
<th>L</th>
<th>2a</th>
</tr>
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<tr>
<td>empirical formulae</td>
<td>C$<em>{26}$H$</em>{15}$N$_3$</td>
<td>C$<em>{127}$H$</em>{99}$F$_6$Fe$_2$N$_7$O$_9$P$_4$Pd$_2$S$_2$</td>
</tr>
<tr>
<td>Fw</td>
<td>369.41</td>
<td>2493.63</td>
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<tr>
<td>Crystal system</td>
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<tr>
<td>Space group</td>
<td>$P$ 21/n</td>
<td>$P$-1</td>
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<tr>
<td>T, K</td>
<td>293(2)</td>
<td>100</td>
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<tr>
<td>$\lambda$ (Mo K$\alpha$), Å</td>
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<td>0.90000 Å</td>
</tr>
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<td>a, Å</td>
<td>17.205(6)</td>
<td>16.949(3)</td>
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<tr>
<td>b, Å</td>
<td>5.746(2)</td>
<td>17.562(4)</td>
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\[
c, \text{ Å} \
20.409(7) \\
\alpha, ^\circ \\
90 \\
\beta, ^\circ \\
104.374(8) \\
\gamma, ^\circ \\
90 \\
V, \text{ Å}^3 \\
1954.4(11) \\
Z \\
4 \\
\rho_{\text{calc}}, \text{ g cm}^{-1} \\
1.255 \\
\mu, \text{ mm}^{-1} \\
0.075 \\
\text{GOF}^a \\
1.192 \\
\text{Final R indices [I>2\sigma(I)]} \\
R1 = 0.1365, wR2 = 0.1480 \\
R1 = 0.0774, wR2 = 0.2297 \\
\text{R indices (all data)} \\
R1 = 0.2732, wR2 = 0.1853 \\
R1 = 0.0920, wR2 = 0.2431 \\
\]

\text{GOF}^a = \left\{ \frac{\sum w(F^2_0 - F^2_c)^2}{(n - p)} \right\}^{1/2}, \text{ where } n \text{ and } p \text{ denotes the number of data points and the number of parameters, respectively.}^b 
\text{R1} = (\Sigma II F_0I - 1F_c II) / \Sigma IF_0I; \text{ }^c wR2 = \left\{ \frac{\sum w(F^2_0 - F^2_c)^2}{\sum w(F^2_0)} \right\}^{1/2}, \text{ where } w = 1/ [\sigma^2(F^2_0) + (aP)^2 + (bP)] \text{ and } P = [\max (0,F^2_0) + 2F^2_c]/3.

\textbf{Table S2: Selected Bonds Distances (Å) and Angles (deg) for L, 2a.}

\begin{tabular}{llll}
\hline
 & N(1)-C(11) & 1.381(8) & N(1)-C(15) & 1.385(8) & N(2)-C(1) & 1.324(13) \\
N(2)-C(5) & 1.306(12) & N(3)-C(24) & 1.326(9) & N(3)-C(25) & 1.317(10) \\
\hline
C(11)-N(1)-C(15) & 110.1(5) & C(1)-N(2)-C(5) & 115.3(8) \\
C(24)-N(3)-C(25) & 114.5(6) & C(11)-N(1)-H(1A) & 128(3) \\
\hline
\end{tabular}
C(15)-N(1)-H(1A)  119(3)  
N(2)-C(5)-C(4)    123.1(9)  
N(1)-C(15)-C(16)  129.3(5)  
N(3)-C(25)-C(26)  125.4(6)  

2a

Pd(1)-N(3B)       2.087(7)  
Pd(1)-N(3A)       2.112(7)  
Pd(1)-P(4)        2.289(3)  
Pd(1)-P(1)        2.305(3)  
Pd(2)-N(1B)       2.056(9)  
Pd(2)-N(1A)       2.101(7)  
Pd(2)-P(2)        2.294(3)  
Pd(2)-P(3)        2.307(3)  

N(3B)-Pd(1)-N(3A)  84.3(3)   
N(3A)-Pd(1)-P(4)   171.6(2)  
N(3A)-Pd(1)-P(1)   87.2(2)   
N(1B)-Pd(2)-N(1A)  85.1(3)   
N(1A)-Pd(2)-P(2)   87.2(2)   
N(1A)-Pd(2)-P(3)   171.6(2)  
N(3B)-Pd(1)-P(4)   90.0(2)   
N(3B)-Pd(1)-P(1)   171.0(2)  
P(4)-Pd(1)-P(1)    98.83(9)  
N(1B)-Pd(2)-P(2)   171.7(2)  
N(1B)-Pd(2)-P(3)   88.3(2)   
P(2)-Pd(2)-P(3)    99.66(10)