## The Synthesis of New 60° Organometallic Subunits and Self-Assembly of Three-Dimensional M<sub>3</sub>L<sub>2</sub>Trigonal-Bipyramidal Cages

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**Figure S1**. (A)  ${}^{1}$ H and (B)  ${}^{13}$ C NMR spectra of **4** in CDCl<sub>3</sub>





**Figure S2.** (A) <sup>1</sup>H and (B) <sup>13</sup>C NMR spectra of **5** in CDCl<sub>3</sub>; (C) <sup>31</sup>P NMR spectrum f **5** in CDCl<sub>3</sub>



**S3** 



**Figure S3**. (A)  ${}^{1}$ H and (B)  ${}^{31}$ P NMR spectra of **6** in CD<sub>2</sub>Cl<sub>2</sub>





**Figure S4**. (A)  ${}^{1}$ H and (B)  ${}^{13}$ C NMR spectra of **7** in CDCl<sub>3</sub>





**Figure S5.** (A)  ${}^{1}$ H and (B)  ${}^{13}$ C NMR spectra of **9** in CDCl<sub>3</sub>



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**Figure S6**. (A) <sup>1</sup>H and (B) <sup>13</sup>C NMR spectra of **10** in CDCl<sub>3</sub>; (C) <sup>31</sup>P NMR spectrum f **10** in CD<sub>2</sub>Cl<sub>2</sub>



**S8** 



С

**Figure S7**. (A)  ${}^{1}$ H and (B)  ${}^{31}$ P NMR spectra of **11** in CD<sub>2</sub>Cl<sub>2</sub>





**Figure S8.** (A) <sup>1</sup>H and (B) <sup>31</sup>P NMR spectra of  $M_3L_2$  TBP cage **13** in Acetone- $d_6/D_2O$ : 1/1





**Figure S9.** (A) <sup>1</sup>H and (B) <sup>31</sup>P NMR spectra of  $M_3L_2$  TBP cage **14** in Dichloromethane- $d_2$ /Acetone- $d_6$ : 7/1







**Figure S10.** (A) <sup>1</sup>H and (B) <sup>31</sup>P NMR spectra of  $M_3L_2$  TBP cage **15** in Dichloromethane- $d_2$ /Acetone- $d_6$ : 7/1





