

## **Tailored Electrochemical Synthesis of 2D-Hexagonal, Lamellar, and Cage-type Mesostructured Pt Thin Films with Extra-large Periodicity**

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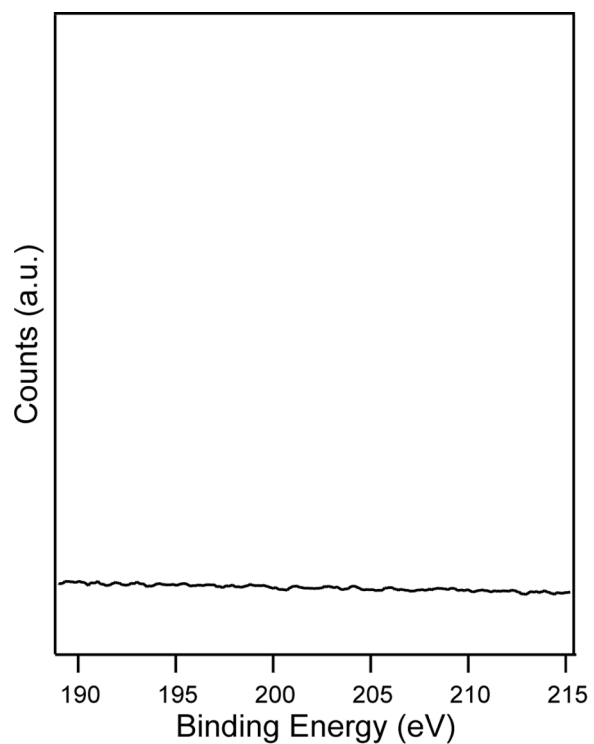
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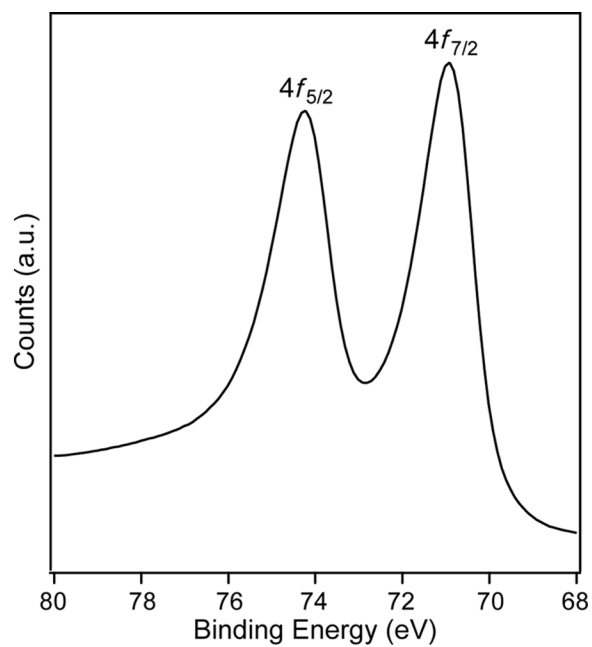
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**Figure S1**



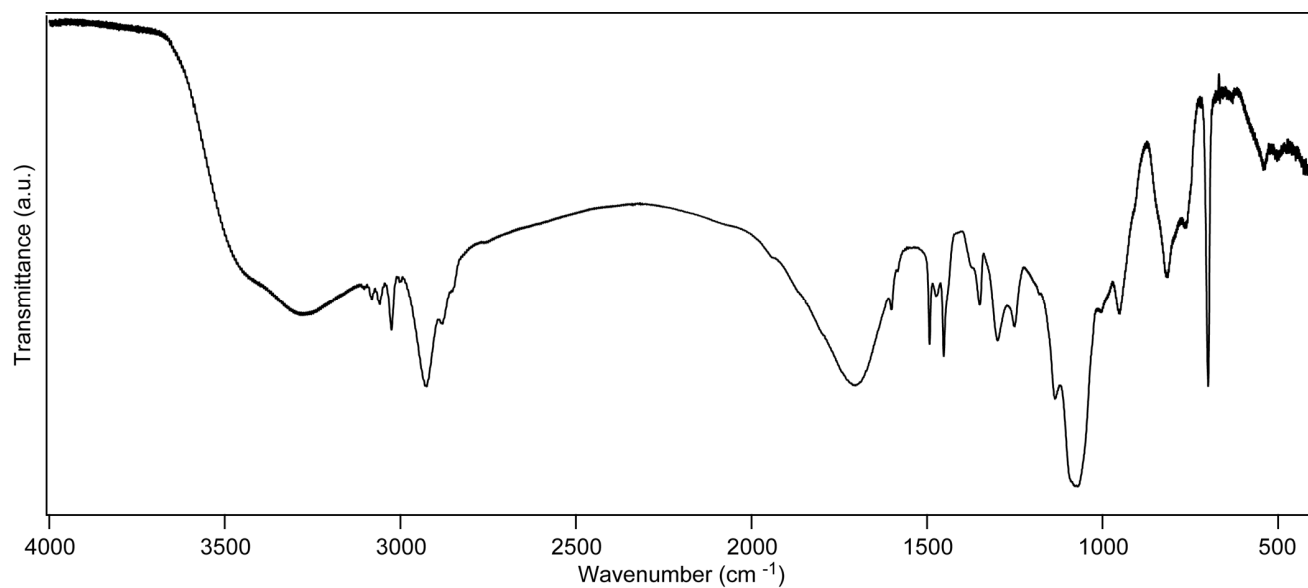
**Figure S1.** XPS spectrum of a mesoporous Pt film prepared from a precursor solution including 0.50 ml of Pt aqueous solution. No peak due to Cl 2p<sub>3/2</sub> is observed around 198 eV which is attributed to chloride ions of H<sub>2</sub>PtCl<sub>6</sub>, indicating that the un-deposited Pt species are removed by washing with THF and water.

**Figure S2**



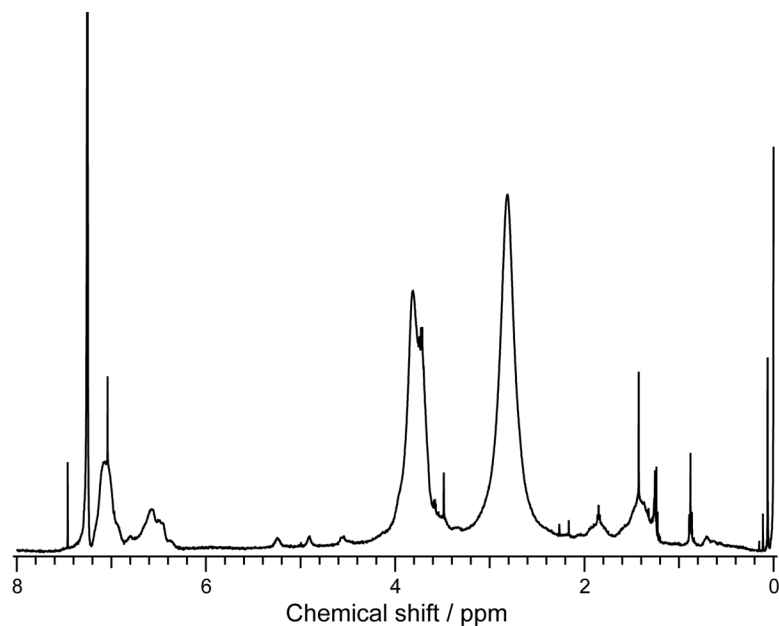
**Figure S2.** XPS spectrum of a mesoporous Pt film with 2D-hexagonal symmetry prepared from a precursor solution including 0.50 ml of Pt aqueous solution.

**Figure S3**



**Figure S3.** IR spectrum of a LLC film prepared from a precursor solution including 0.50 ml of Pt aqueous solution. The characteristic absorption band around 1079 cm<sup>-1</sup> assigned to C-O stretching vibration of EO groups is slightly shifted to lower energy, which is an evidence for the formation of hydrogen bonding between EO groups and Pt aqua complexes.<sup>33</sup>

**Figure S4**



**Figure S4.** Liquid-state  $^1\text{H}$  NMR spectrum of LLC films prepared from a precursor solution including 0.50 ml of Pt aqueous solution. The characteristic signals due to THF at  $\delta = 1.8$  and 3.7 ppm are not observed in the spectrum, proving the evaporation during the LLC formation (the amount of remaining HF is under the limit of detection).