**Supplementary Information:** MOF-derived PtCo/Co₃O₄ nanocomposites in carbonaceous matrices as high-performance ORR electrocatalysts synthesized *via* Laser Ablation techniques

**LASiS Experimental Set-up**

The LASiS experimental arrangement utilized for fabrication of all the NCs in this work primarily consisted of a high-energy laser-source along with an in-house-built jacketed-reactor cell, as schematically represented in Fig. S1. Specifically, a Q-switched Nd:YAG pulsed laser source was utilized (Manufacturer: Brilliant Inc.; Model: Brilliant Easy) to provide a laser beam with wavelength of 1064 nm and 4ns long pulses with an energy of 330 mJ per pulse at a repetition rate of 10 Hz. The generated unfocused beam was directed to the surface of a Co metal target, which in turn, was immersed in the desired liquid solution. The metal target-liquid pair were contained in the interior of the sealed stainless steel jacketed-reactor cell during the entire procedure. A high damage threshold tested laser window was used to seal reaction-cell, while the temperature in the core of the cell could be precisely determined through a connected-thermostat.

![Figure S1 – Schematic LASiS experimental set up](image)

**High-temperature hydrothermal digestion process**

All the ICP-OES specimen were initially digested using a high-temperature hydrothermal process. Specifically, 5 mg of the desired sample was dispersed in 10 mL of concentrated a HNO₃ solution. The resulting dispersion was sealed in a 50 mL Teflon-lined autoclave and held at 200 °C for 6 hours. The resulting solution was then collected and further dilute with deionized water to yield a final volume of 40 mL.
Supporting Crystalographic, Morphological and Functional analyses

Figure S2 – TEM micrographs of ZIF-67 structures produced via LASIS.

Figure S3 – Slow-scan XRD of 125, 93 and 65-PtCo@C-Co$_x$O$_4$ NCs in the Pt-Co (111) diffraction region
Figure S4: SEM micrographs of ZIF-67 synthesized in (a) absence of K₂PtCl₆; and (b) presence of 125 mg/L K₂PtCl₆.

Figure S5: Cyclic voltammetry curves performed at a scan rate of 100 mV/s in a N₂-saturated 1M KOH solution.
Table S1: Summary of (i) characteristic 2θ Pt-Co (111) diffraction peak; (ii) calculated Pt-Co (111) lattice constant; (iii) estimated Pt:Co alloying ratio from Vegard’s law; and (iv) weight percentage of Pt for samples 125, 93 and 65-PtCo@C-Co₃O₄

<table>
<thead>
<tr>
<th>Sample</th>
<th>2θ Pt-Co (111)</th>
<th>Lattice Constant (Å)</th>
<th>Pt:Co (Vegard’s law)</th>
<th>Pt (%Weight)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>125-PtCo@C-Co₃O₄</td>
<td>40.27*</td>
<td>3.876</td>
<td>7.388</td>
<td>5.20 %</td>
</tr>
<tr>
<td>95-PtCo@C-Co₃O₄</td>
<td>40.14*</td>
<td>3.888</td>
<td>9.499</td>
<td>4.42 %</td>
</tr>
<tr>
<td>63-PtCo@C-Co₃O₄</td>
<td>40.13*</td>
<td>3.889</td>
<td>9.713</td>
<td>4.16 %</td>
</tr>
</tbody>
</table>

*The determination of Pt (%Weight) was based on the following expression:

\[%W_{Pt} = \left( \frac{m_{Pt}}{m_{NC}} \right) \times 100\]

Where \(m_{Pt}\) denotes the mass of Pt present in each sample (determined via ICP-OES) and \(m_{NC}\) indicates the total mass of analyzed LASiS-synthesized NC.