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Supplementary Information

Metal–Organic Frameworks Derived Co_xFe_{1-x}P Nanocubes for Electrochemical

Hydrogen Evolution

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Figure S1 FESEM images and EDX images of Fe PB (A), Co PB (B), PBAs-3-1 (C), PBAs-3-2 (D), and

PBAs-3-4 (E).



Figure S2 TEM images of Fe PB (A), Co PB (B), PBAs-3-1 (C), PBAs-3-2 (D), and PBAs-3-4 (E).



Figure S3 (A) XRD patterns of MOFs template: Fe PB (a), Co PB (b), PBAs-3-1 (c), PBAs-3-2 (d), PBAs-3-4 (e). (B) XRD patterns after phosphidation: FeP (a), CoP (b), $Co_{0.71}Fe_{0.29}P$ (c), $Co_{0.59}Fe_{0.41}P$ (d), and $Co_{0.38}Fe_{0.62}P$ nanocubes (e).



Figure S4 FESEM images of FeP (A) and CoP (B).



Figure S5 TEM images of FeP (A) and CoP (B) at different magnification.



Figure S6 N 1s XPS spectrum of $Co_{0.71}Fe_{0.29}P$ (A), $Co_{0.59}Fe_{0.41}P$ (B), and $Co_{0.38}Fe_{0.62}P$ nanocubes (C)



Figure S7 EDX images of $Co_{0.71}Fe_{0.29}P$ (A), $Co_{0.59}Fe_{0.41}P$ (B), and $Co_{0.38}Fe_{0.62}P$

nanocubes (C).



Figure S8 N₂-sorption isotherms of $Co_{0.71}Fe_{0.29}P$ (A), $Co_{0.59}Fe_{0.41}P$ (B) and $Co_{0.38}Fe_{0.62}P$ nanocubes (C).



Figure S9 N₂-sorption isotherms of PBAs-3-1 (A), PBAs-3-2 (B), and PBAs-3-4 (C).



Figure S10 Cyclic voltammograms (CVs) of $Co_{0.71}Fe_{0.29}P$ (A), $Co_{0.59}Fe_{0.41}P$ (B) and $Co_{0.38}Fe_{0.62}P$ nanocubes (C). Current density differences (Δj) plotted against scan rates

(D). Δj is the difference between anodic and cathodic current densities at potential indicated by the black dash lines, where no redox current peaks are observed. The linear slopes in (D) are equivalent to twice of the electrochemical double-layer capacitances (C_{dl}). C_{dl} is used to represent and compare the apparent electrochemical surface area (ECSA) between Co_{0.71}Fe_{0.29}P (A), Co_{0.59}Fe_{0.41}P (B) and Co_{0.38}Fe_{0.62}P nanocubes because of their similar composition.



Figure S11 SEM image of the $Co_{0.59}Fe_{0.41}P$ after cycling test. No obvious morphology changes can be found when compared to the system before the tests.



Figure S12 Polarization curves of MOFs templates compare with $Co_{0.59}Fe_{0.41}P$ nanocubes in 0.5 M H₂SO₄ (A) at a scan rate of 5 mV s⁻¹ and 1.0 M KOH (B) at a scan rate of 2 mV s⁻¹.



Figure S13 XRD patterns of $Co_{0.59}Fe_{0.41}P$ nanocubes prepared at 350 °C (a), 450 °C

(b), and 550 °C (c).



Figure S14 FESEM images of $Co_{0.59}Fe_{0.41}P$ nanocubes prepared at 350 °C (A), 450

°C (B), and 550 °C (C) at different magnification.



Figure S15 TEM images of $Co_{0.59}Fe_{0.41}P$ nanocubes prepared at 350 °C (A), 450 °C (B), and 550 °C (C) at different magnification.