Supplementary Information

## Intercalation chemistry and thermal characteristics of layered double hydroxides possessing organic phosphonates and sulfonates

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Fig. S1 FE-SEM images characterizing LDH-CO<sub>3</sub> (bottom) and LDH-2-heP (top).



Fig. S2 Aqueous solutions containing LDH-2-heP (top left), LDH-2-heS (top right), LDH-3-hpS (bottom left), and LDH-1,3-pDS (bottom right).



**Fig. S3** XRD patterns characterizing LDH-**2-heS** (a), a sample obtained by heating LDH-**2-heS** to 300 °C in air (b), LDH-**2-heP** (c), samples obtained by heating LDH-**2-heP** in air to 200 °C (d) and 300 °C (e).



**Fig. S4** XRD patterns characterizing the following LDH samples heated at 500 °C under the specified gas atmosphere: a, LDH-**2-heS** in 10% H<sub>2</sub> in helium; b, LDH-**3-hpS** in 10% H<sub>2</sub> in helium; c, LDH-**1,3-pDS** in N<sub>2</sub>; d, LDH-**1,3-pDS** in 10% H<sub>2</sub> in helium; e, LDH-**2-heP** in N<sub>2</sub>; f, LDH-**2-heP** in 10% H<sub>2</sub> in helium.



**Fig. S5** TG profiles characterizing LDH-**CO**<sub>3</sub> (dotted line) and LDH-**3-hpS** samples prepared at various **3-hpS**/A1 ratios: dash-dotted line, 0.05; dashed line, 0.5; solid line, 1.5.