Figure S1: Raman spectra (a) and XRD patterns (b) of the solid phase obtained at the end of the Mg$^{2+}$ solution titration.

Figure S2: Titration curves for different $x_{\text{solution}}$ for (a) Ni$^{2+}$-Fe$^{3+}$ mixed cation solutions and (b) Mg$^{2+}$-Fe$^{3+}$ mixed cation solutions.

Figure S3: (a) Raman spectra of Ni$^{II}$-Fe$^{III}$ LDH with different $x_{\text{layer}}$ values and (b) correlation between the wavenumber of the LDH Raman band and the $x_{\text{layer}}$ values. The numbers in brackets refer to the standard deviation for each parameter of the linear regression.
Figure S4: (a) Raman spectra of Mg²⁺-Fe³⁺ LDH with different $x_{layer}$ values and (b) correlation between the wavenumber of the LDH Raman band and the $x_{layer}$ values. The numbers in brackets refer to the standard deviation for each parameter of the linear regression.

Figure S5: Raman spectra in the spectral range 100-1200 cm⁻¹ (a) same as Figure 8a and (b) same as Figure 12a