

Supporting Information for

High-temperature redox chemistry
of $\text{La}_{1.5+x}\text{Sr}_{0.5-x}\text{Co}_{0.5}\text{Ni}_{0.5}\text{O}_{4+\delta}$ ($x = 0.0, 0.2$) studied *in situ* by
neutron diffraction

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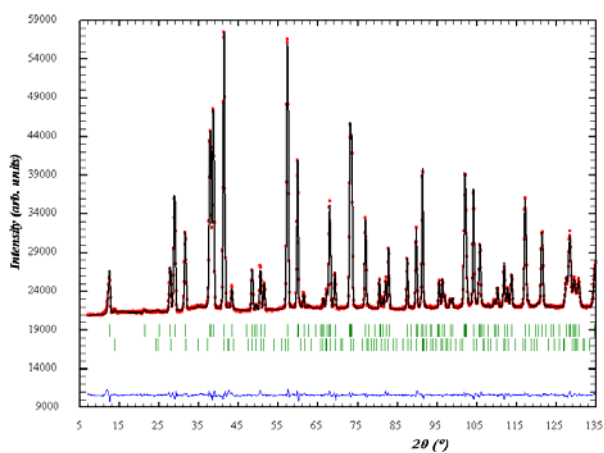
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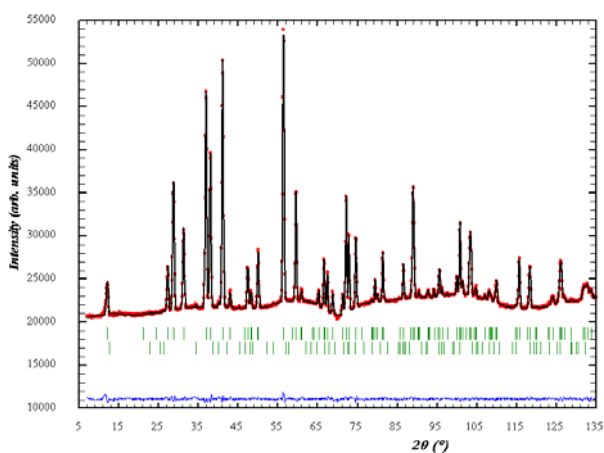
Figure S1. Rietveld refinements of the neutron powder diffraction patterns of $\text{La}_{1.7}\text{Sr}_{0.3}\text{Co}_{0.5}\text{Ni}_{0.5}\text{O}_{4+\delta}$ ($x = 0.2$) **(a)** at 43 °C before hydrogen reduction, **(b)** at 600 °C after reduction, **(c)** at 57 °C after reduction. The lower set of tick marks correspond to a $\text{La}(\text{OH})_3$ impurity at 43 °C and a La_2O_3 impurity at 600 and 57 °C. The undulating background in (b) and (c) is due to diffuse scattering from the quartz tube.

Figure S2. Difference Fourier map at $z = 0.25$ showing the presence of an interstitial anion in as-prepared $\text{La}_{1.5}\text{Sr}_{0.5}\text{Co}_{0.5}\text{Ni}_{0.5}\text{O}_{4+\delta}$.

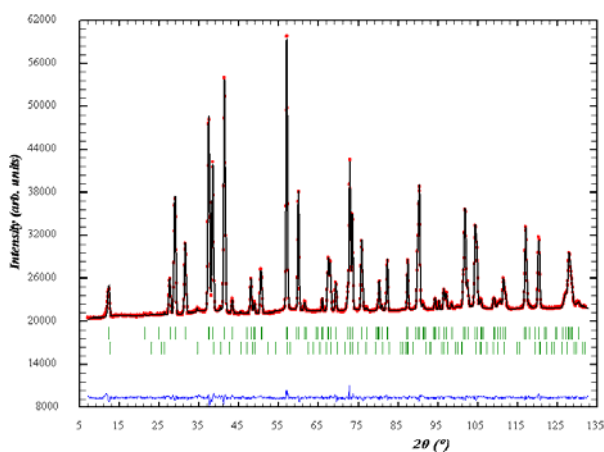
Figure S3. Difference Fourier map at $z = 0.25$ showing the presence of an interstitial anion at room temperature in H_2 -reduced $\text{La}_{1.7}\text{Sr}_{0.3}\text{Co}_{0.5}\text{Ni}_{0.5}\text{O}_{3.90(1)}$.



(a)



(b)



(c)

Figure S1

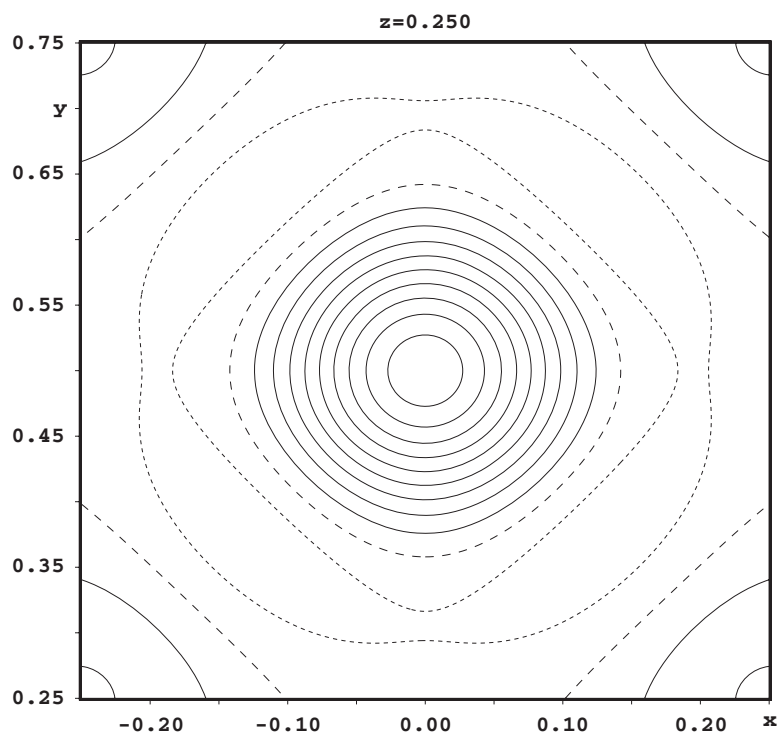


Figure S2

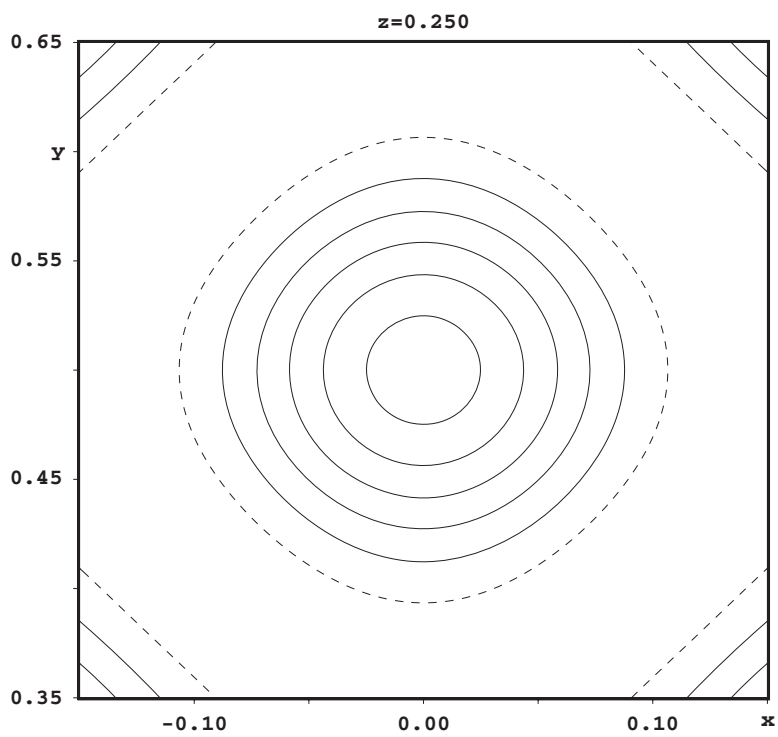


Figure S3