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Supporting information for article:

In situ neutron diffraction to investigate the solid-state synthesis of Ni-rich cathode materials

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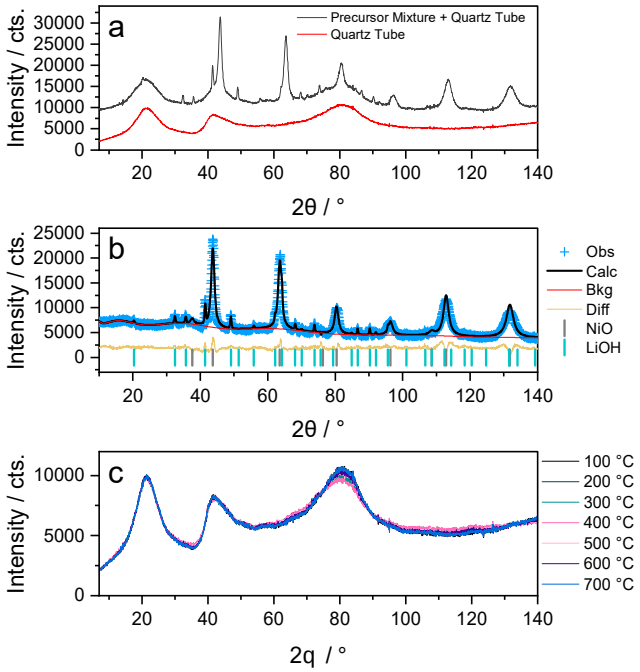

Figure S1 a) Raw data collected from the initial pattern of the LiOH + NiO heating experiment and an empty quartz tube. b) Example of Rietveld refinement profile for the initial pattern of the LiOH + NiO heating experiment after subtraction of the quartz tube background profile. c) Evolution of diffraction patterns collected from an empty quartz tube at various temperatures.

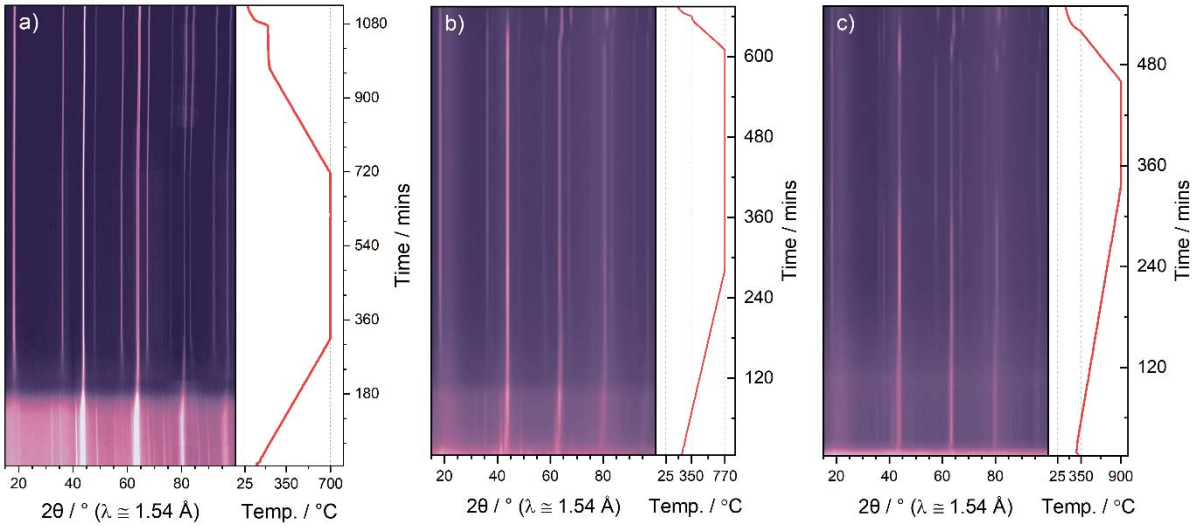

Figure S2 Contour plots of diffraction data collected during heating and then cooling of the three mixtures targeting a) $LiNiO_2$, b) $LiNi_{0.9}Mn_{0.1}O_2$ and c) $LiNi_{0.75}Mn_{0.25}O_2$.

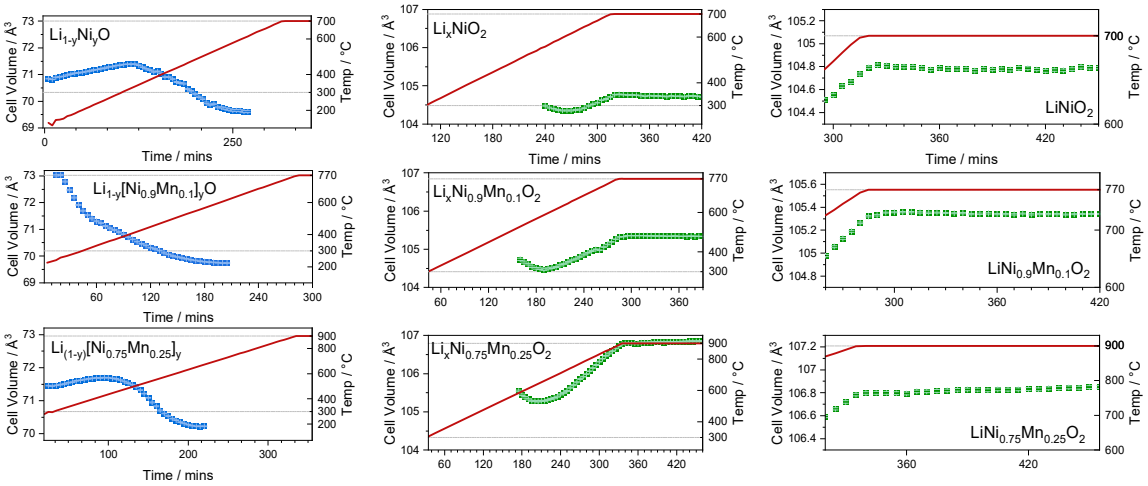

Figure S3 Unit cell volume of the cubic (left) and layered phases (middle, right) as a function of time during the heating experiments.

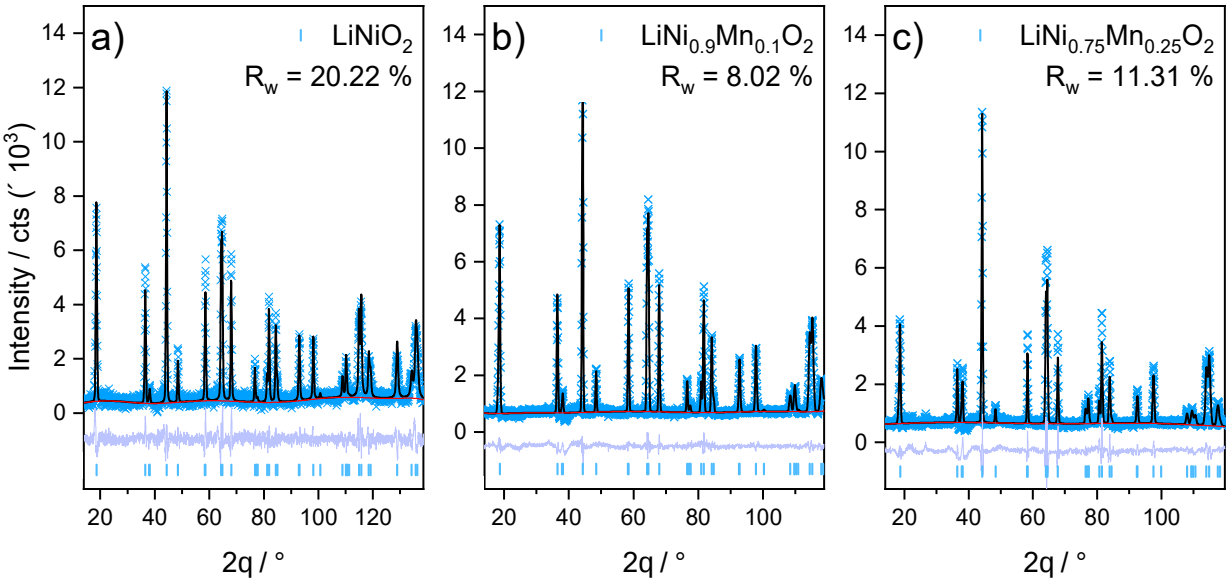

Figure S4 Rietveld refinement profiles of the synthesised materials: a) $LiNiO_2$, b) $LiNi_{0.9}Mn_{0.1}O_2$ and c) $LiNi_{0.75}Mn_{0.25}O_2$ after cooling to 200 °C.

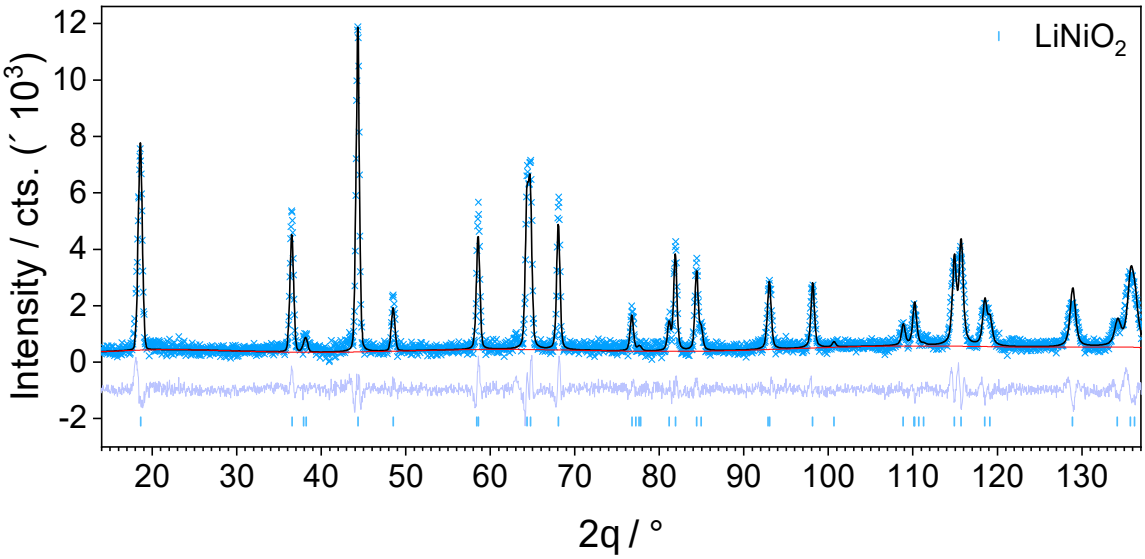

Figure S5 Rietveld refinement profile of the synthesised $LiNiO_2$ phase after cooling to ambient temperature.

Table S1 Structure parameters and atomic co-ordinates of the synthesised $LiNiO_2$ phase after cooling to ambient temperature.

Atom	**Site**	**Sym.**	**x**	**y**	**z**	**frac**	**U_{iso} / Å^2**
Li1	3b	-3m	0	0	0.5	0.981(6)	0.00165
Ni1	3b	-3m	0	0	0.5	0.019(6)	0.00165
Li2	3a	-3m	0	0	0	0.019(6)	0.00788
Ni2	3a	-3m	0	0	0	0.981(6)	0.00788
Mn2	3a	-3m	0	0	0	0.000	0.00788
O	6c	3m	0	0	0.25797(27)	1.000	0.00877
Space group = $R\bar{3}m$, R_w = 20.97 %, R_F = 7.69 % a = 2.8737(2) Å, c = 14.1831(9) Å, Vol = 101.432(8) Å^3							