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Supporting information for article:

Pitfalls and reproducibility of *in situ* synchrotron powder X-ray diffraction studies of solvothermal nanoparticle formation

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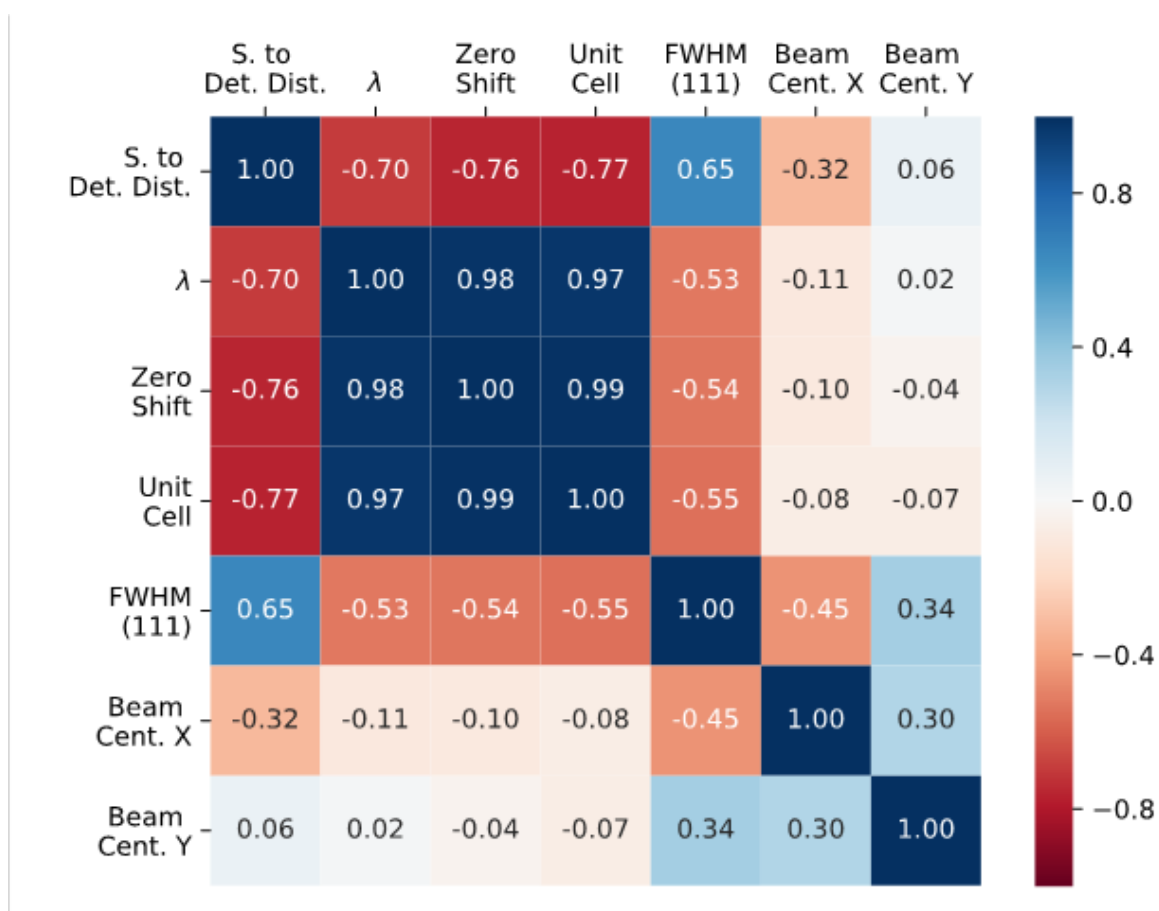
Spearman correlation map

Figure 1: Spearman correlation map of calibrated and refined parameters from the LaB₆ synchrotron PXRD datasets. A coefficient of 1 indicates a complete positive correlation, 0 is no correlation and -1 means complete inverse correlation.

In situ PXRD data contour plots

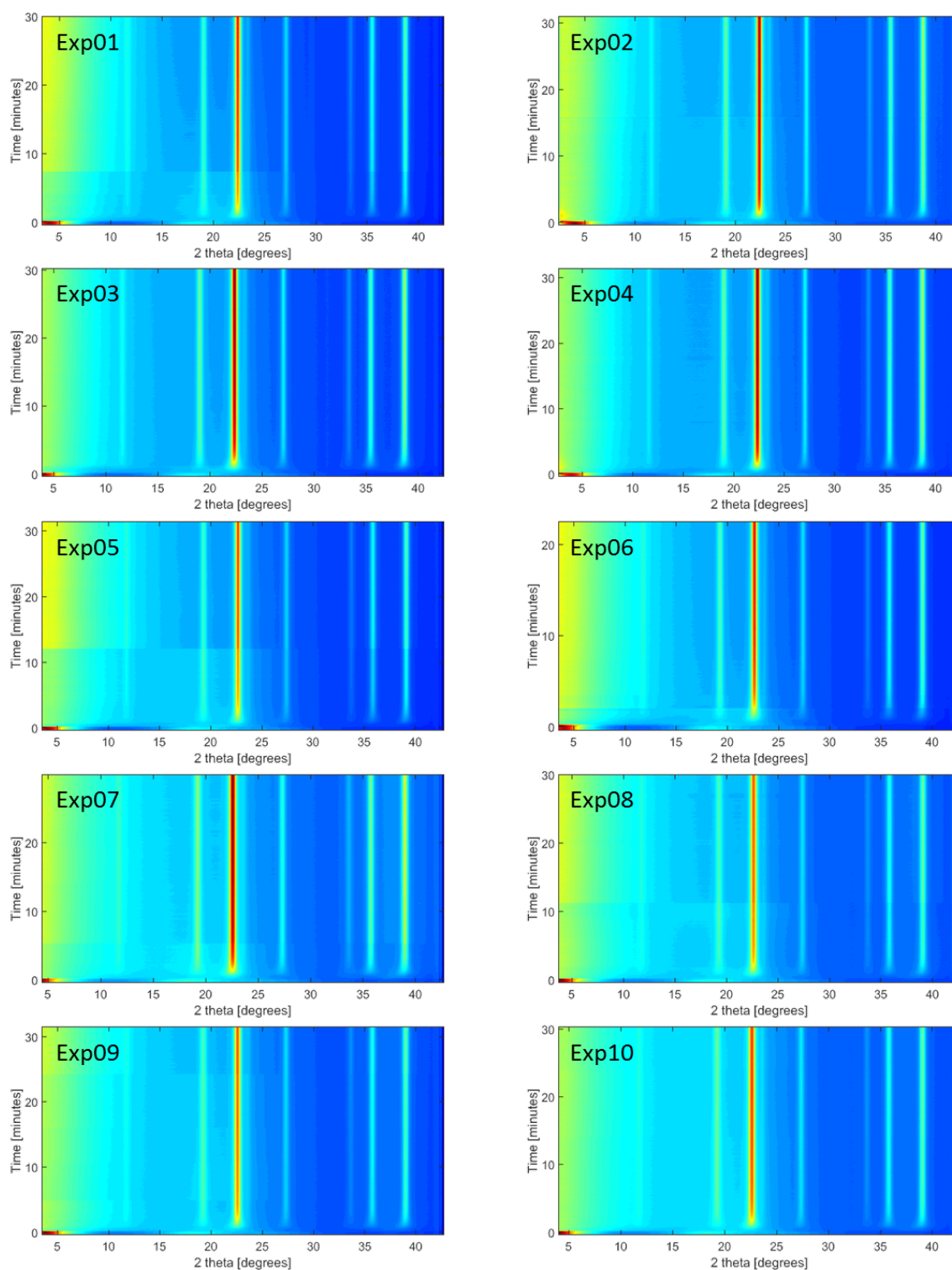


Figure S2: *In situ* PXRD data contour plots for the ten reproducibility experiments on the hydrothermal formation of γ -Fe₂O₃ nanocrystallites at 300 °C and 250 bar. In the plots, red corresponds to the maximum intensity and dark blue corresponds to the lowest.

Representative Rietveld refinements

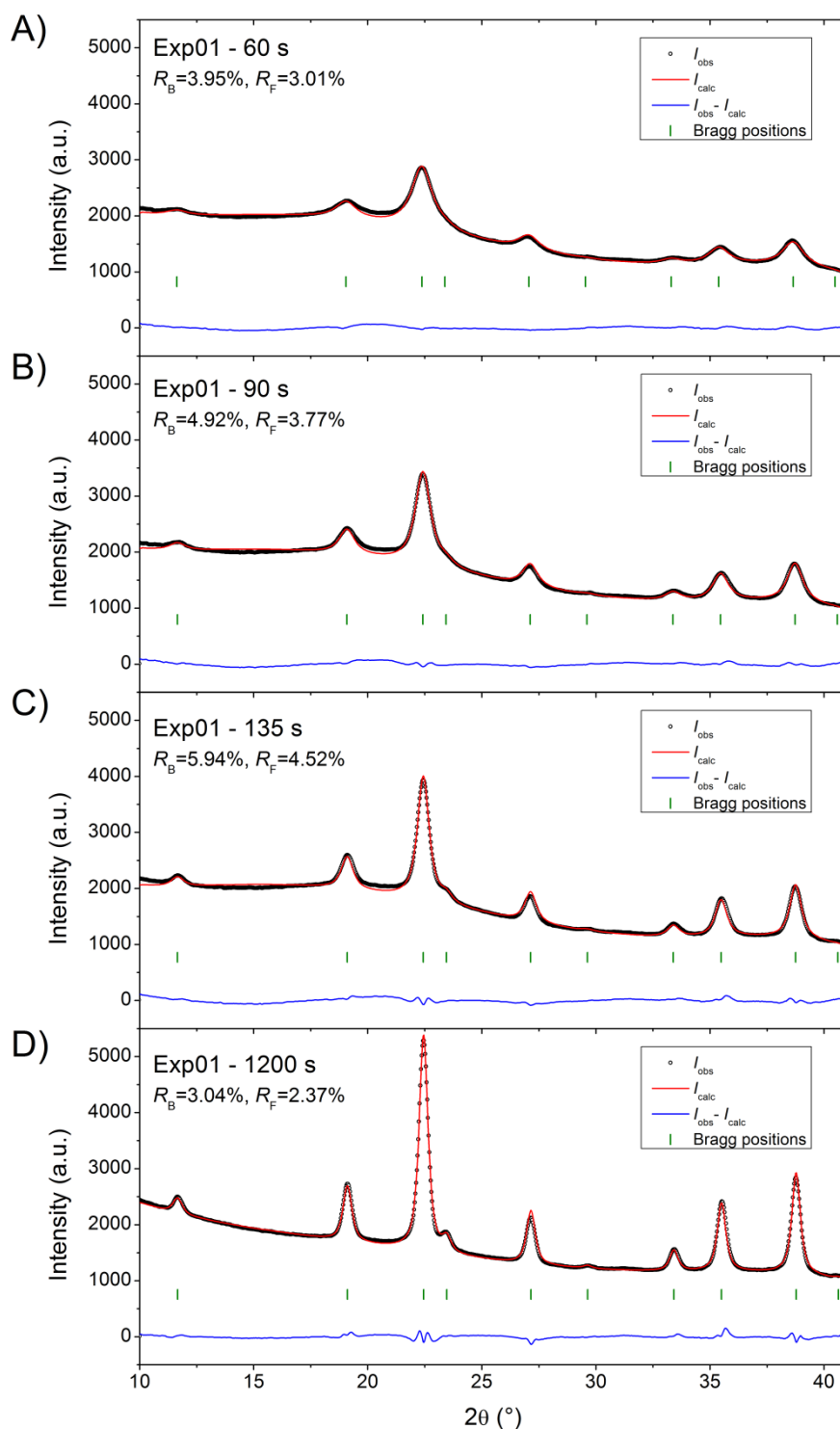


Figure S3: Representative *in situ* synchrotron PXRD patterns and corresponding Rietveld refinements after A) 60 seconds, B) 90 seconds, C) 135 seconds, and D) 1200 seconds of experiment 1.

Representative WPPM refinements

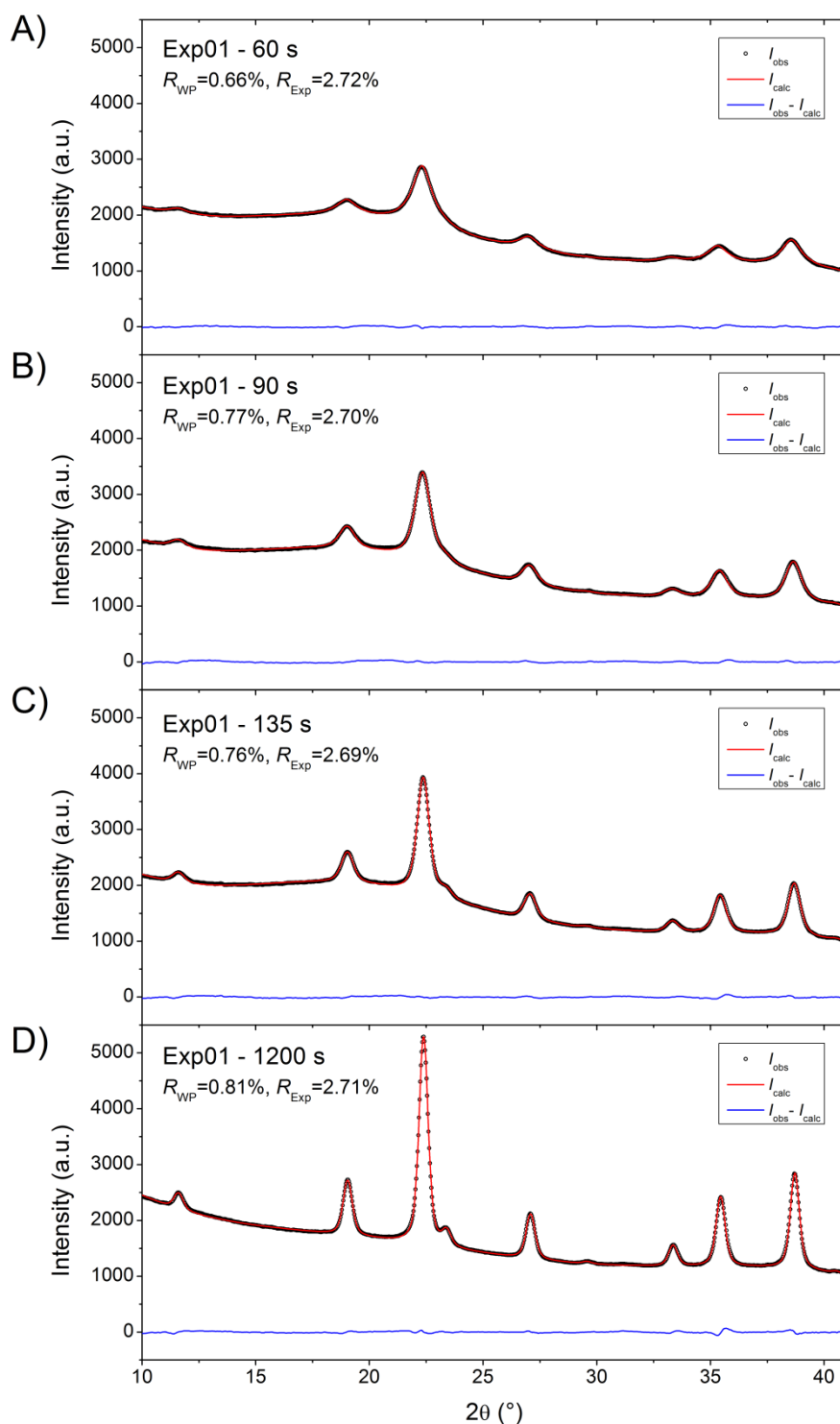


Figure S4: Representative *in situ* synchrotron PXRD patterns and corresponding WPPM models after A) 60 seconds, B) 90 seconds, C) 135 seconds, and D) 1200 seconds of experiment 1.