# Resolving fast relative kinetics of inorganic materials from *in situ* studies

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Solid-state synthesis is traditionally performed at high temperatures in a muffle furnace to overcome the limited ion mobility in the solid state. However, these conventional approaches are often operated ‘behind a closed furnace door’, making solid-state synthesis a black box problem. Product formation, potential side reactions, byproducts, and impurities can only be assessed after cooling the reaction mixture to room temperature, often hours, days, or weeks later. These high-temperature synthesis procedures favour the formation of thermodynamically stable products, potentially missing short-lived intermediates or metastable phases that may form intermittently with unique properties.

Despite the blindness of these reactions to the transformations that occur, *in situ* synchrotron X-ray powder diffraction has been utilised to gain mechanistic insight into solid-state reactions. Based on the expectation that the reactions are slow and occurring over multiple hours or days, these *in situ* synchrotron experiments typically use small sample sizes and progressive heating to track reaction progression as a function of temperature. However, recent studies have suggested that some solid-state reactions occur much faster, often on the order of a few seconds [1].

Simple solid-state reactions will be presented, with the mechanism of their transformations observed using a custom-designed reactor to track solid-state syntheses as a function of both temperature and time [2]. The earliest stages of solid-state reactions are observed, showing the formation of metastable phases with ground state energies above the convex hull that can then be trapped and cooled to room temperature [3]. By understanding the early stages of these solid-state reactions, mechanisms can be proposed that lead to eventual product and phase selectivity.

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[2] Hu, D., Beauvais, M.L., Mullens, B.G., Sanchez Monserrate, B.A., Vornholt, S.M., Kamm, G.E., Ferrari, J.J., Chupas, P.J., Chapman, K.W. (2023), *J. Appl. Cryst.*, **57**.

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