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Modular Reactor for In Situ X-ray Scattering, Spectroscopy, and ATR-IR Studies of Solvothermal Nanoparticle Synthesis

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Modular Reactor for In Situ X-ray Scattering, Spectroscopy, and ATR-IR Studies of Solvothermal Nanoparticle Synthesis

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We describe a novel reaction reactor for in situ X-ray scattering and spectroscopy with liquid or gas injection capabilities. The reactor enables autoclave-like conditions for solvothermal synthesis, with heating up to 200 ℃ at pressures of up to 8 bar, as well as cooling to −20 ℃ to decelerate rapid reduction processes such as those occurring during cluster formation. Optional ATR IR or injection can be performed simultaneously by the choice of different caps for the inlet. While conventional ex situ techniques usually provide information only on the final products, the underlying reaction mechanisms often remain obscured, potentially missing crucial intermediate steps. However, a deeper insight into the reaction step is of great interest to tailor material properties, especially for those at the nanoscale.[1],[2],[3] To shine a light on these mechanisms, our multi-purpose in situ cell standardises reaction procedures across a wide range of non-invasive monitoring methods with a strong focus on high flux synchrotron radiation. Capabilities of the cell to fetch complexation and cluster intermediates have been demonstrated for Cu3PdN, ZnS and Fe3O4 nanoparticles.[4],[5],[6]

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