Crystallization of Polyethylene Glycol in Polyethylene Glycol – Sucrose Solutions During Freeze-Thawing and In Situ Freeze-Drying
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NNIN Facility utilized: Characterization Facility

Objectives
- To study the phase behavior in frozen PEG and PEG–Sucrose solutions during freeze-thawing
- To improve the sensitivity of the XRD signal during in situ freeze-drying for quantification of PEG crystallization during freezing and drying stages of lyophilization

Freeze-thawing of PEG: Sucrose Solutions
2% w/v PEG + 2% w/v Sucrose 10% w/v PEG + 10% w/v Sucrose

PEG peaks disappeared at ~-15°C similar to the \( T_{\text{eutectic melt}} \) observed using Differential Scanning Calorimetry (~-16°C).

Results
- The XRD signal was substantially enhanced with a 950 \( \mu \text{L} \) sample volume during in situ freeze-drying; thus enabling quantification of PEG crystallization during freezing, primary, and secondary drying.