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CryoSAXS as a Method for Measuring Low Resolution Macromolecular Structure

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Small angle X-ray scattering (SAXS) is an increasingly popular technique for obtaining low resolution structural information from macromolecules and complexes in solution. Biomolecular SAXS signals can rapidly degrade due to radiation damage, so that flow or oscillating cells and large total sample volumes may be required. For particularly sensitive or hard to produce samples, such as of light sensitive proteins, metalloenzymes, and large complexes, and studies where multiple buffer conditions are probed sample consumption may be prohibitive. We describe cryo-cooling of samples to 100 K to prevent X-ray induced radiation damage. We identify SAXS-friendly cryoprotectant conditions that suppress ice formation upon cooling, and compare cryoSAXS profiles obtained in window-free variable-path-length cells with room temperature measurements for a variety of standard molecules. We obtain data sufficient for envelope reconstructions using scattering volumes as small as 20 nL, and find good agreement between cryoSAXS data and known atomic structures. We also discuss work on developing low-volume fixed path-length sample holders for cryoSAXS. Cryo-cooled samples can withstand doses that are 2-3 orders of magnitude higher than typically used for SAXS at room temperature, comparable to those used in cryo-crystallography. While practical challenges remain, cryoSAXS opens the possibility of studies exploiting high brightness X-ray sources and mail-in high-throughput SAXS. This work is funded by the NSF (DBI-1152348).

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