Xtalab Synergy-ED: To Cryo, Or Not to Cryo, That Is the ED Question

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The Rigaku XtaLAB Synergy-ED is a fully integrated electron diffractometer with a seamless workflow from data collection to 3D structure determination. The XtaLAB Synergy-ED is the result of Rigaku's collaboration with JEOL, synergistically combining each partner's core technologies: Rigaku's hybrid-pixel electron detector (HyPix-ED) and CrysAlisPro software, and JEOL's long-standing excellence in electron beam generation and control.
Using 3D electron diffraction (3DED), a.k.a. microED, single crystals of all classes below one- micron in size can be studied. The XtaLAB Synergy-ED offers a wide range of experiments, including low temperature, which confers benefits such as: reduced beam damage; improved resolution; reduced disorder; and in the case of electron diffraction, stabilization of samples in vacuo. As electron diffraction requires samples to be studied under high vacuum, cryo-transfer - freezing of samples prior to introduction to vacuum - is essential for sensitive compounds, e.g. proteins or MOFs. Combining cryo-transfer with variable detector distance further allows the study of solvated crystals, including proteins, in the XtaLAB Synergy-ED.
Here, we report two small molecule examples. Both are supramolecular solids featuring thymine (T) and 2,6-diaminopurine (D) in different stoichiometries (2:1 and 3:2). Thermal analyses (TGA-DSC) show that both samples are hydrated co-crystals. 3DED experiments at room temperature and high vacuum resulted in their anhydrous species (see Figure, left).
Cryo-transfer of flash-cooled crystals of the same batch measured at 100 K under high vacuum gave the 2:1 T-D monohydrate co-crystal and 3:2 T-D trihydrate co-crystal, respectively (see Figure, right). Calculated powder patterns from 3DED data match the experimental PXRD data, confirming phase purity. Our work unambiguously demonstrates that cryo-transfer techniques used with the XtaLAB Synergy-ED enabled the determination of hydrated structures, despite high vacuum conditions.

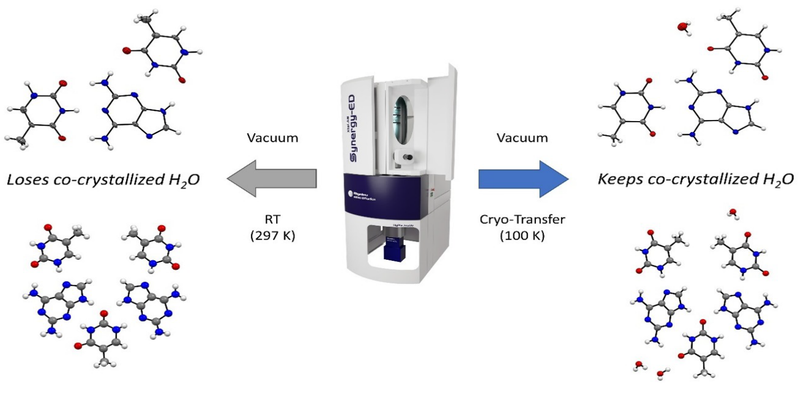

Figure 1 Two examples illustrating cryo-transfer is important: (left) structures measured at ambient temperature and vacuum, and (right) using a cryo-transfer station at 100 K.

Figure 1