Challenges and Victories: Variable Temperature MicroED on Small Molecules

M Laurent LOOS (Rigaku Europe SE, NEU ISENBURG)
Lee DANIELS (Rigaku Americas Corp, The Woodlands, TX, USA)
Khai TRUONG (Rigaku Europe SE, NEU-ISENBURG - Allemagne)
Robert BÜRKER (Rigaku Europe SE, NEU-ISENBURG - Allemagne)
Christian GÖB (Rigaku Europe SE, NEU-ISENBURG - Allemagne)
Christian SCHÜRMANN (Rigaku Europe SE, NEU-ISENBURG - Allemagne)
Mathias MEYER (Rigaku Polska, WROCLAW - Pologne)

Abstract

Since our launch of the XtaLAB Synergy-ED in 2021, users have produced hundreds of new and re-visited structures via MicroED, including over 500 unique structures from Rigaku application labs alone. Most of the early studies were conducted at ambient temperature. We commonly perform low- or variable-temperature experiments, allowing more in-depth study of crystalline materials that are sensitive to vacuum and/or electron-beam damage, and that undergo temperature-accessible phase transitions.

Since the Synergy-ED goniometer is compatible with many of the cryo-holders already available for TEM instruments, structural scientists may now use MicroED at non-ambient temperatures, including variable-temperature studies.

Cryo-transfer specimen holders such as the Gatan ELSA provide protection of samples before introduction to the vacuum, allowing the study of solvates and other vacuum-sensitive species. The programmable temperature control also allows exploration of phase-transition behavior. We will discuss results from samples for which cryo-transfer proved essential.

The Hummingbird Scientific MEMS biasing/heating holder offers the possibility to increase temperature, allowing for exploration of the phase behavior of samples such as porous materials. Our recent results using single-crystal data from electron diffraction on a MOF system, Cu(ta)2 (Hta = 1H-1,2,3-triazole), at room temperature and at 200 °C, were compared to a previous study of the same material from 2012 done using SC-XRD and PXRD.