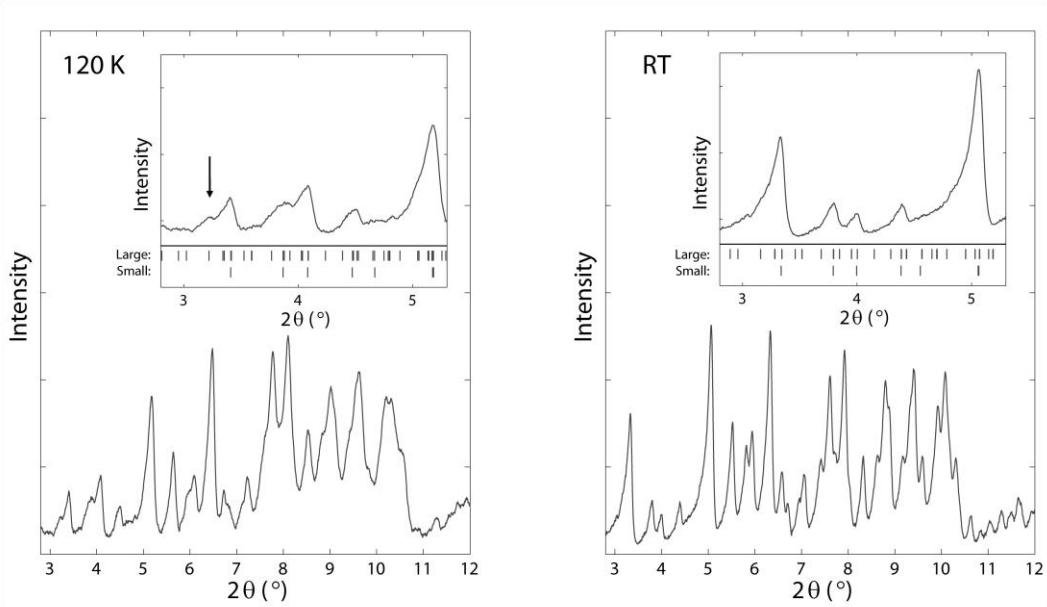
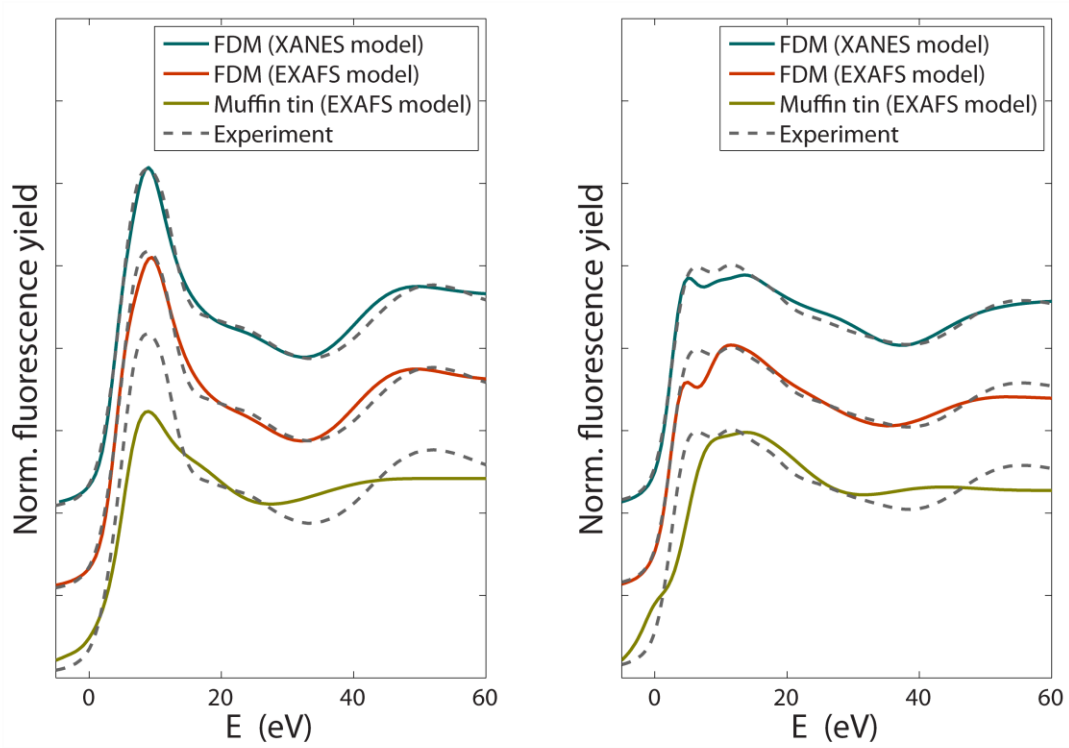
Supplementary Material



Supplementary Figure 1. XRPD patterns of R_6 insulin collected at 120 K (*a*) and RT (*b*). Insets: magnification of the low angle region (2θ = 3−5º) and the positions of the Bragg peaks for the large ($a \approx 160$ Å, c ≈ 80 Å) and small (a ≈ 80 Å, c ≈ 40 Å) unit cell, respectively. The peak pointed out at 3.25º in the cold spectrum indicates that the cold phase must have doubled unit cell parameters.



Supplementary Figure 2. XANES calculated on a 4.5 Å cluster around each Zn-atom of (*a*) T_6 insulin, and (*b*) R_6 insulin. Calculation were done on the input model (EXAFS model) using both the MT approximation (green) and FDM approach (red), and on the model optimized by *FitIt* (XANES model) using the FDM approach (blue). The calculated spectra are compared with experimental XANES (dashed). The offset of the energy scale is 9659 eV corresponding to the *K*-edge position of metallic zinc.