



**Figure S1** - Plots of Rietveld refinement of the original (subcell) structures for  $\text{Tl}_2(\text{SC}_6\text{H}_4\text{S})$  (top) and  $\text{Tl}_2(\text{SeC}_6\text{H}_4\text{Se})$  (bottom). Data were collected in capillaries on material diluted with ground cork. Blue points are the measured data, the red curve is that calculated from the structural model with the difference shown below in black. Tick marks indicate allowed peak positions.